

## APPENDICES

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# **UIC PERMIT RENEWAL APPLICATIONS**

**FOR**

**SPL-1 (PERMIT NUMBER IN-127-1W-0001)  
WAL-1 (PERMIT NUMBER IN-127-1W-0003)  
WAL-2 (PERMIT NUMBER IN-127-1W-0004)**

**for  
BETHLEHEM STEEL CORPORATION, INC.  
BURNS HARBOR DIVISION**

**OCTOBER 1997**

**VOLUME 2 OF 2**

**TEXAS WORLD OPERATIONS INC  
520 POST OAK BLVD SUITE 450  
HOUSTON TEXAS 77027-9405  
(713) 850-0003**



## APPENDIX B

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, DC 20460

EPA

## COMPLETION FORM FOR INJECTION WELLS

## ADMINISTRATIVE INFORMATION

## 1. Permittee

Midco Remedial Corporation

Address (Permanent Mailing Address) (Street, City, State, and ZIP Code)

c/o Karaganis & White, Ltd.  
414 Orleans Street, Suite 810  
Chicago, Illinois 60610

## 2. Operator

Environmental Resources Management-EnviroClean-North Central, Inc.

Address (Street, City, State, and ZIP Code)

540 Lake Cook Road, Suite 300  
Deerfield, Illinois 60015

## 3. Facility Name

Midco I &amp; II Superfund Sites Waste Disposal Well #1

## Telephone Number

(219) :944-8571

Address (Street, City, State, and ZIP Code)

7400 West 15th Avenue  
Gary, Indiana 46406


## 4. Surface Location Description of Injection Well(s)

State Indiana		County Lake	
1/4 of NE	1/4 of SW	1/4 section NW Section 11	
Township 36 North		Range 9 West	
Feet from (N/S) 1,109		Line of quarter section and	
Feet from (E/W) 750		Line of quarter section	

Submit with this Completion Form the attachments listed in Attachments for Completion Form.

## CERTIFICATION

I certify under the penalty of law that I have personally examined and am familiar with the information submitted in this document and all attachments and that, based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the information is true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment. (Ref. 40 CFR 144.32).

NAME AND OFFICIAL TITLE (Please type or print) Ron E. Hutchens, P.E. Project Manager	SIGNATURE 	DATE SIGNED 5/1/97
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## 6.0 FORMATION STRESS TEST

The formation stress test (i.e., a "mini-frac" test), which was performed on a portion of the Lower Mount Simon Sandstone, will allow the USEPA to set the upper limit of injection pressure that can be applied to that formation. The test results are included in Appendix I, and summarized in this section.

The Midco Deep Well No. 1 was drilled approximately 30' into the Lower Mount Simon Sandstone (i.e., to a depth of about 2,657'). Subsequently, the entire Mount Simon "B" Cap was cased off, leaving the uppermost 30' of the Lower Mount Simon Sandstone to be completed as a 9-7/8" diameter open-hole completion. After setting the casing, the hole was advanced to 2,810' with a 6-1/4" diameter bit. At this point, a string of DST tools were run in the hole, and the dual compression packers were set to enable the performance of the formation stress test in the interval from 2,711.2' to 2,810' (i.e., a 98.8' gross interval) in the 6-1/4" diameter hole. In addition to the usual DST tools, equipment required for the test included:

- A high-resolution, quartz, capacitance-type pressure gauge. The gauge was equipped with a surface readout capability to constantly monitor the downhole pressure, and was positioned by using an electric wireline at 2,660'.
- In-line flow meters. These meters allowed an accurate measurement of flow rates from 1/4 to 10 bpm.

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- Suction and discharge filters. These filters were installed on the pump truck that was used to filter the injection fluid (i.e., Gary city water) down to 5 microns.
- An oxygen scavenger. The scavenger Halliburton "Fe-5" (a reducing agent) was mixed with the injection fluid at the pump truck to ensure that no iron-based precipitates would accumulate in the formation because of the injected fluid.

With the surface equipment installed, the pressure gauge in place, and the initial pressure readings stabilized, injection began at 25.6 gallons per minute (gpm). This rate was held constant for 30 minutes to establish a constant bottom hole pressure. Next, the rate was increased to 51.2 gpm. At this flow rate, the bottom hole pressure increased substantially (i.e., up to 1,988 psi or a gradient of 0.747 psi/ft). Therefore, it was necessary to cut back the flow rate to 39.9 gpm after 11 minutes, and back to 23.1 gpm after an additional 10 minutes. This flow rate was held constant for 42 minutes to allow the reservoir to reestablish a baseline isobar of 1,488 psi. Afterwards, the injection flow rate was again increased to 51.7 gpm and held constant for one hour and 18 minutes to establish a constant injection pressure. Incremental, flow-rate increases were then performed to bring the flow rate to 63, 73.5, 84.8, 105, and 126 gpm. At this point, a one-hour fall-off test was initiated to secure data for future analysis if required. The flow-rate steps and associated bottom hole pressures are presented in Table 6-1.

Appendix I contains the majority of the formation stress test data, which were included in a report submitted by Halliburton. There may be slight discrepancies in data points where the Halliburton pump truck varied from a constant rate for a very short period

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of time, and several minutes were required to again attain the desired constant rate. These data have been smoothed over by hand calculation.

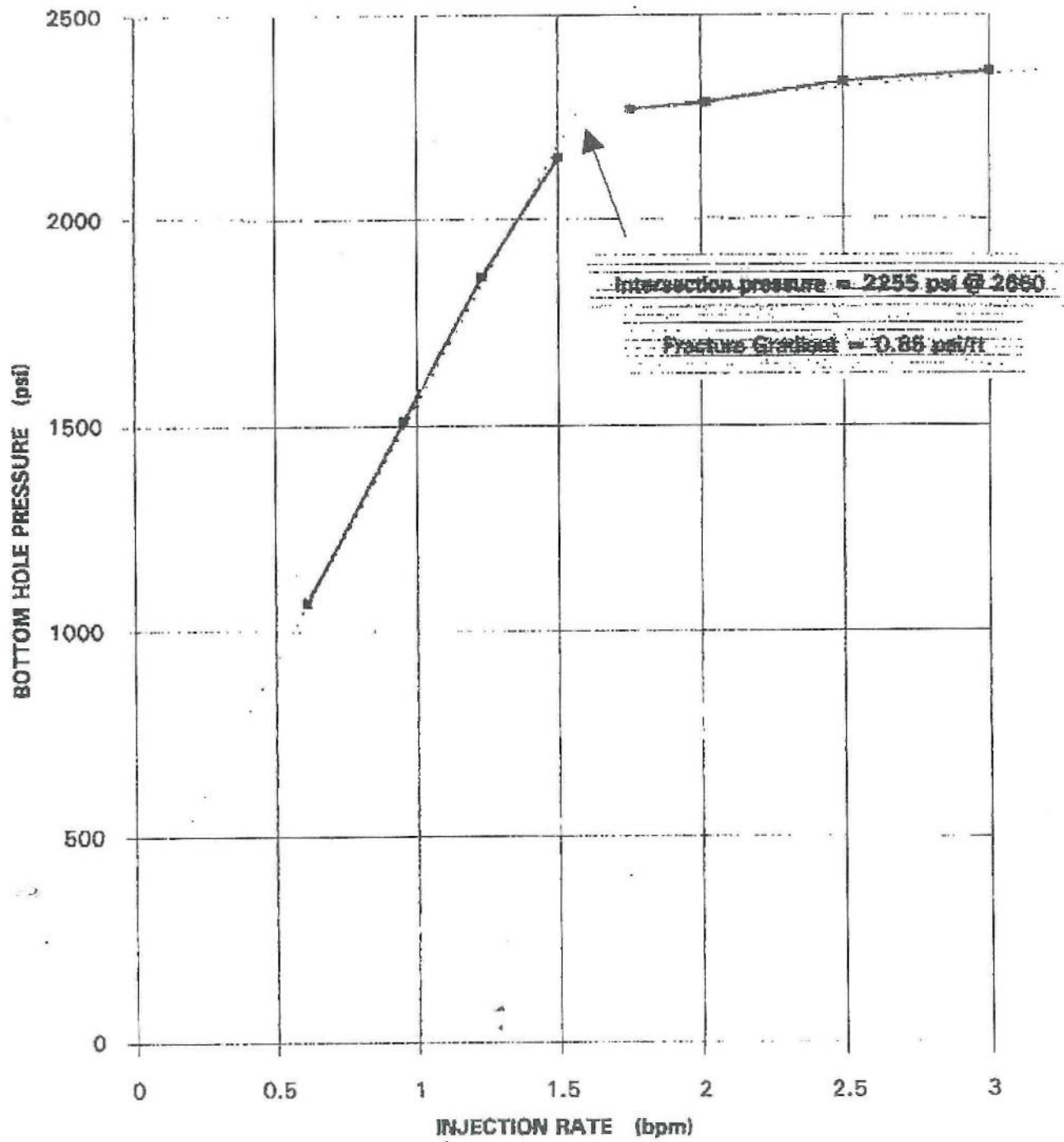
A plot of bottom hole pressure versus flow rate is illustrated in Figure 6-1. The intersection of the two distinct lines developed on the graph is the fracture pressure of that part of the Lower Mount Simon Sandstone that was subjected to the stress test. Although the pressures are measured at 2,660', the fracture pressure gradient is applicable at the depth of concern.

The resulting fracture gradient of 0.85 psi/ft is considered high for this formation but is within the range of 0.55 to 0.9 psi/ft. established in the literature. The data collected during the mini-frac test indicate that the Lower Mount Simon Sandstone is capable of receiving the planned injection rate of less than 100 gpm without fracturing. The raw pressure and time data obtained from the test are recorded on a 3-1/2" computer disc in ASCII format and presented in Appendix I of the USEPA and IDEM copies of this report.

TABLE 6-1

BOTTOM HOLE PRESSURES  
RECORDED DURING FORMATION STRESS TEST  
MIDCO I DEEP WELL NO. 1  
MIDCO SUPERFUND SITE  
GARY, INDIANA

Rate (gpm)	Bottom Hole Pressure (psi)
25.6	1070
39.9	1509
51.7	1861
63.0	2150
73.5	2270
84.8	2286
105.0	2337
126.0	2362

**FIGURE 6-1****Stress Test Results**

## APPENDIX C

BETHLEHEM STEEL CORPORATION  
BURNS HARBOR DIVISION

WASTE ANALYSIS PLAN  
for  
UIC Permit Nos. IN-127-1W-0001, 0003 and 0004

APPENDIX C

EFFECTIVE: October 1997

October, 1997

Bethlehem Steel Corporation  
Burns Harbor Division

WASTE ANALYSIS PLAN FOR  
UIC Permit Nos. IN-127-1W-0001, 0003 and 0004

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Attachment 1 - Facility Map

Attachment 2 - Waste Ammonia Liquor Appendix IX Analysis

Attachment 3 - Spent Pickle Liquor Appendix IX Analysis

Attachment 4 - Sample Label, Sample Seal and Chain of Custody Form

October, 1997

Bethlehem Steel Corporation  
Burns Harbor Division

WASTE ANALYSIS PLAN FOR  
UIC Permit Nos. IN-127-1W-0001, 0003 and 0004 (cont.)

Table 1 - Analytical Methods for Waste Analysis

Table 2 - Constituents and Historic Concentrations

Table 3 - Detection Limits (DL)/Practical Quantitation Limits  
(PQL)/Reporting Limits (RL)

Appendix 1 - Standard Operating Procedures

- A. Chromium
- B. Lead
- C. Selenium
- D. Volatile Organics
- E. Semi-Volatile Organics
- F. Specific Conductivity
- G. Specific Gravity
- H. Total Dissolved Solids
- I. Total Organic Carbon

October, 1997

## **SECTION I**

### **SCOPE AND APPLICABILITY**

As required by Federal Regulation 40 CFR 146.68(a), this waste analysis plan describes the wastes that are disposed via deepwell injection as well as the procedures that will be followed to obtain a detailed chemical and physical analysis of a representative sample of the waste streams.

## **SECTION II**

### **RESPONSIBLE CHARGE AND PLAN ADMINISTRATION**

The Superintendent of the Division's Environmental Services Department is in charge of this Waste Analysis Plan (WAP). His responsibilities, which may be carried out by qualified persons under his supervision, include the following.

1. Periodic review of the WAP in order to ensure its continued applicability.
2. Ensure that all required analyses are performed.
3. Recordkeeping of analytical results and quality assurance.

## **SECTION III**

### **DESCRIPTION OF WASTE STREAMS**

The Burns Harbor Division currently operates three Class I deepwells for the disposal of two hazardous waste streams. Two of the wells (Permit Nos. IN-127-1W-0003 and IN-127-1W-0004) dispose of Waste Ammonia Liquor (WAL), which is characteristically hazardous (toxicity characteristic) for the constituents selenium, benzene and pyridine. One of the wells (Permit No. IN-127-1W-0001) disposes of Spent Pickle Liquor, which is a listed hazardous waste (K062). The following provides a description of how each waste stream is generated, stored and transported prior to disposal.

#### **A. Waste Ammonia Liquor (WAL)**

WAL is generated at the Coke Oven Coal Chemical Plant, which is located in the southwest corner of the Burns Harbor Division (See Attachment 1 - Facility Map). WAL from coking operations (a coal carbonization process) is a wastewater that is generated from "... extraction, beneficiation and processing of ores and minerals (including coal)" [40 CFR 261.4(b)(7)].

Coal is the principal input into the coking operation and coke, coal tar and coke oven gas are the principal outputs. Ammonia liquor is recirculated in a closed-loop recycle system to cool and cleanse the coke oven gas produced by the coking process. The water, tars and solids are scrubbed from the gas flow to one of three decanters where phase separation is allowed to occur. The Ammonia liquor floats on the top of the coal tar and overflows a fixed weir that discharges to a holding tank. The majority of the ammonia liquor is recirculated from the holding tank to the coke oven gas main as flushing liquor for further gas cooling and

cleaning. Excess water is introduced into the closed-loop system primarily as a result of the liberation of moisture, which is contained within the coal, during the coking process. In order to maintain proper liquid equilibrium within the closed-loop system, the excess or waste ammonia liquor (WAL) is discharged to the WAL system for processing and disposal.

The WAL is initially processed through the WAL Clarifier for tar and sediment removal through gravimetric settling. The overflow from the clarifier is discharged to spiral cooler heat exchangers in order to lower the temperature of the WAL from approximately 140 degrees to 80 degrees Fahrenheit. After cooling, the WAL is pumped to one of three pressure filters for initial filtration. Following this initial filtration, the WAL is directed to one of four polishing filters for the removal of fine particulates. After final filtering, the WAL is directed to one of the two injection wells for final disposal.

#### B. Spent Pickle Liquor (SPL) K062

SPL is generated as a by-product of the steel sheet pickling operation, which is located in about the center of the Burns Harbor Plant (See Attachment 1 - Facility Map). The continuous pickling operation uses a hydrochloric acid solution in a cascading (e.g., counter-current flow) system to remove scale and rust from the steel strip in preparation for further processing in the cold mill.

The hydrochloric acid solution is maintained at a 6.5 percent concentration in tank number 3 and is heated using steam. The acid flows toward tank number 1 as it loses strength. The acid in tank number 1 is at about a 1.5 percent concentration and overflows to a 35,000-gallon storage tank, at which point it is considered to be SPL. The SPL from this tank can be transferred to the Secondary Wastewater Treatment Plant for use as a wastewater treatment chemical, transferred to a truck loading station for sale as a substitute for a commercial chemical product or transferred to the SPL well system for processing and disposal by deep well injection. If the SPL is to be deep well injected, the SPL from the storage tank is pumped using the filter feed pumps to a pressure filtration system for solids removal. The SPL then flows through a heat exchanger for temperature adjustment prior to flowing to the deep well for disposal.

## SECTION IV

### WASTE CHARACTERIZATION

#### A. Waste Ammonia Liquor

Attachment 2 contains a copy of an Appendix IX analysis performed on WAL in October 1993. Based on this analysis as well as other hazardous waste analyses performed, WAL consistently exceeds the Toxicity Characteristic Leaching Procedure (TCLP) maximum concentration specified in 40 CFR 261.24, Table 1 for Selenium, Benzene and Pyridine. In addition, the WAL injected contains less than one percent suspended solids. Therefore, under the TCLP procedure, the WAL is analyzed on a total constituent basis. Based on this information, the following parameters will be analyzed under this WAP at the frequency indicated.

<u>Parameter</u>	<u>Frequency of Analysis</u>
0010 Selenium, Total	Monthly
0010 Benzene, Total	Monthly
0030 Pyridine, Total	Monthly
pH	Continuous*
Specific Conductance	Monthly
Specific Gravity	Monthly
Temperature	Continuous*
Total Dissolved Solids	Monthly
Total Organic Carbon	Monthly

Guidance #8  
~~Ex~~  
 >0.01% by mass

\*Temperature and pH are monitored continuously, except during periods of instrument maintenance and calibration, and recorded on strip chart recorders at the Waste Ammonia Liquor Pump Station. Grab samples are obtained five days per week (i.e., Monday through Friday) to verify calibration of the instruments.

#### B. Spent Pickle Liquor

Attachment 3 contains a copy of an Appendix IX analysis that was performed on SPL during October, 1993. When SPL is disposed as a waste, it is a listed hazardous waste with an EPA hazardous waste number K062. Based on 40 CFR 261, Appendix VII, the specific constituents for which SPL is listed as hazardous are hexavalent chromium and lead. Based on this information, the following parameters will be analyzed under this WAP at the frequency indicated.

<u>Parameter</u>	<u>Frequency of Analysis</u>
Chromium, Total	Monthly
Lead, Total	Monthly
Specific Conductance	Monthly
Specific Gravity	Monthly
Temperature	Continuous*
Total Dissolved Solids	Monthly
Total Organic Carbon	Monthly

40 CFR 269.43  
 add H<sub>2</sub>S  
 Guidance #8  
 pH, ~~Ex~~  
 >0.01% by mass

\*Temperature is monitored continuously, except during periods of instrument maintenance and calibration, and recorded on strip chart recorders at the Spent Pickle Liquor Pump Station.

The above waste characterization sampling frequencies and parameters are justifiable on the basis that the constituent concentrations for the injected wastes have been consistent since monitoring began.

## SECTION V

### WASTE ANALYSIS PLAN QUALITY ASSURANCE/QUALITY CONTROL

#### A. Description of Sampling

##### 1. Sampling Methods for Waste Ammonia Liquor

Samples of WAL will be obtained at the frequency specified in Section IV of this WAP. Samples will be obtained from the sample tap located at the Waste Ammonia Liquor Pump Station. This line taps off of the main injection line after all filter devices and immediately prior to the valve that directs the WAL to one of the two well heads for injection.

The samples will be collected into clean, pre-preserved, labeled sample bottles supplied by the analytical laboratory. In order to obtain the samples, the sample collector will open the sample tap and allow WAL to flow from the tap for at least 30 seconds. He will fill each of the supplied sample bottles with WAL without immersing the sample tap line into the WAL sample. When each bottle is full, the sample collector will seal that bottle, place it into the sample cooler, and begin to fill the next bottle. This will continue until all of the sample bottles have been filled. Upon completion of sampling event, the sample collector will record the sample date, time and his initials on each sample bottle's label, complete a sample bottle seal and place it on each sample bottle and complete the chain of custody form (see Attachment 4 for examples of the sample label, sample seal and chain of custody form). The sample collector will then place ice and the completed chain of custody form (sealed in a plastic bag) into the cooler, and transport the cooler to the designated pick-up location for the analytical laboratory.

## 2. Sampling Methods for Spent Pickle Liquor

Samples of SPL will be obtained at the frequency specified in Section IV of this WAP. Samples will be obtained from the drain valve of the 35,000-gallon spent pickle liquor storage tank located west of the spent pickle liquor deepwell control room. This valve will provide samples of SPL immediately prior to filtration and injection.

The samples will be collected from the drain pipe using a dedicated long-handled polyethylene dipper. The sample collector will open the drain valve and allow SPL to flow from the drain line for at least 30 seconds. He will then rinse the dipper at least three times with the SPL flowing from the drain line. After rinsing, he will collect the sample and transfer it from the dipper into clean, pre-preserved, labeled sample bottles supplied by the analytical laboratory. When each sample bottle is full, the sample collector will seal that bottle, place it into the sample cooler and begin to fill the next bottle. This will continue until all sample bottles have been filled. Upon completion of sampling events, the sample collector will record the sample date, time and his initials on each sample bottle's label, complete a sample bottle seal and place on each sample bottle and complete the chain of custody form (see Attachment 4 for examples of the sample label, sample seal and chain of custody form). The sample collector will then place ice and the completed chain of custody form (sealed in a plastic bag) into the cooler, and transport the cooler to a designated pick-up location for the analytical laboratory.

Results of analyses performed in accordance with this plan will be kept on file by the Environmental Services Department for a minimum of five (5) years.

## 3. Sampling Information

- i. Sample Collector - RWR Technical Services, Inc.  
7852 West State Road 2  
LaPorte, IN 46350-9468
- ii. Sampler's Title - Technician
- iii. Sample Collection Method - Refer to Section  
V.A.a. & b. 1 + 2
- iv. Sample Collection Point - Refer to Section  
V.A.a. & b.  
1 + 2

- v. Sample Preservation - To be provided by the analytical laboratory, as needed.
- vi. Sample Frequency - Refer to Section IV
- vii. Analytical Method for Parameter Detection/Quantification - Refer to Table 1.
- viii. Anticipated Analytical Method Accuracy - Refer to Table 4
- ix. Anticipated Upper and Lower Analytical Method Quantification Limit - Refer to Table 3
- x. Adequate Field Documentation of Sampling - Refer to Section V. B. d.

## B. Description of Analytical Methods

### 1. Equipment Cleaning Blanks

The WAL samples will be collected directly from a sample tap into clean, pre-preserved and labeled sample bottle supplied by the analytical laboratory. The SPL will be collected from a drain pipe using a dedicated long handled polyethylene dipper. Therefore, cross-contamination is eliminated and decontamination of the sampling equipment is not necessary.

### 2. Trip Blanks

Each time a group of sample bottles is prepared for use in the WAL sampling event, a 40 ml VOA trip blank bottle will be included in each shipping container which contains environmental samples for VOA analysis. The VOA trip blank bottles are filled with deionized, analyte free water by the laboratory. The trip blanks will be transported to the sampling location and returned (unopened) to the laboratory in a manner identical to the handling procedure used for the environmental samples. These trip blanks will be analyzed for the same VOA parameters identified in Section IV as the WAL samples. If contaminants are found in the blanks, the source of the contamination will be identified and corrective action will be initiated.

### 3. Duplicate Samples and Field Blanks

- Duplicate Analysis: Duplicate samples will be obtained during each sampling event. One duplicate sample per sampling event will be collected and analyzed for the same parameters identified in Section IV.

- Field Blanks: BSC plans to utilize trip and field blanks to aid in assuring and evaluating the quality of data obtained through the sampling program and laboratory analytical procedures.

### 4. Sample Chain of Custody Protocol

Samples will be collected following the above sampling procedures. The sampling times and dates, sample description, sample field data (pH, temperature and injection pressure), volume and number of containers will be noted. Sample identification will be assigned prior to sample collection.

Samples will be accompanied by a properly completed chain of custody form. The sample identification and locations will be listed on the chain of custody form. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date and note the time on the record.

For analyses that must be performed at an off-site laboratory, samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis, with a signed chain of custody record enclosed in each sample box or cooler.

#### 5. Equipment Calibration

Calibration of laboratory equipment will be based on written procedures. Records of calibration, repairs, or replacement will be filed and maintained by laboratory personnel performing quality control activities. These records will be filed at the location where the work is performed. For all instruments, the laboratory will maintain a factory trained repair staff with in-house spare parts or will maintain service contracts with vendors.

#### 6. Data Reduction / Data Validation

The laboratory will perform analytical data reduction and validation under the direction of the Laboratory QA Officer. The Laboratory QA Officer is responsible for assessing data quality and advising of any data which were rated "preliminary" or "unacceptable" or other notations which would caution the data user of possible unreliability. Data reduction, validation, and reporting by the laboratory will be conducted following the general procedures below, although internal laboratory procedural documentation will take precedence:

- Raw data produced by the analyst is turned over to the respective area supervisor.
- The area supervisor reviews the data for attainment of quality control criteria as outlined in the project guidelines and/or established U. S. EPA methods, and for overall reasonableness.
- Upon acceptance of the raw data by the area supervisor, a computerized report is generated and sent to the Laboratory QA Officer.
- The Laboratory QA Officer will complete an audit of reports at a frequency of at least one in twenty, and an audit of every report for consistency.
- The QA Officer and area supervisors will decide whether any sample reanalysis is required.
- Upon acceptance of the preliminary reports by the QA Officer, final reports will be generated.

Data reduction reporting procedures will be those specified in the SW-846 method for inorganic and organic analyses.

The laboratory will prepare and retain full analytical and QC documentation. Such documentation need not be hard (paper) copy, but may be in other storage media (e. g. magnetic tape). As needed, the laboratory will supply a hard copy of the retained information.

Laboratory detection limits and/or quantitation limits for analytical data produced are contained in Table 3. Data on reporting limits and method detection limits can be found in the individual Standard Operating Procedures (SOPs) in Appendix 1. The reporting limit for each chemical parameter will also be indicated on the analytical reports provided to BSC and regulatory agencies

by the laboratory conducting the analysis. The data in Table 3 assumes that there are no severe interferences, and that sample dilutions are not required due to excessive concentrations of target analytes.

#### 7. Internal Quality Control

There will be two types of quality assurance used to ensure the production of analytical data of known documented usable quality: program quality assurance and analytical method quality control.

The analytical laboratory will have a written Quality Assurance / Quality Control program which provides rules and guidelines to ensure the reliability and validity of work conducted at the laboratory. Compliance with the QA/QC program will be coordinated and monitored by the laboratories' Quality Assurance Department, which is independent of the operating departments.

The objectives of the laboratory QA/QC Program are to:

- Ensure that all procedures are documented, including any changes in administrative and/or technical procedures.
- Ensure that all analytical procedures are conducted according to sound scientific principles and have been validated.
- Monitor the performance of the laboratory by a systemic inspection program and provide for corrective action as necessary.
- Collaborate with other laboratories in establishing quality levels, as appropriate.
- Ensure that all data are properly recorded and archived.

All procedures are documented in writing as either Standard Operating Procedures (SOPs) or Methods of Procedures (MP) which are audited and controlled by the QA Department. Internal quality control procedures for analytical services will be conducted by the laboratory in accordance with their SOP and the individual method requirements in a manner consistent with appropriate SW-846, 40 CFR Part 136, or other validated analytical methods. These specifications include the types of audits required (sample spikes, surrogate spikes, reference samples, controls, blanks), the frequency of each audit, the compounds to be used for sample spikes and surrogate spikes, and the quality control acceptance criteria for these audits.

The laboratory will document, in each data package provided, that both initial and ongoing instrument and analytical QC functions have been met. Any samples analyzed in non-conformance with the QC criteria will be reanalyzed by the laboratory, if sufficient sample volume is available.

#### 8. Laboratory Audits

The QA Manager will perform internal laboratory audits. The

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system audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain of custody procedures, sample preparation and analysis, instrument operating records, etc. The performance will be conducted on an annual basis.

#### 9. Corrective Actions

Corrective actions will be implemented when any aspect of the analytical or sampling method does not achieve the project objectives. This may entail re-sampling and/or re-analyzing (for a particular parameter) the wastestreams, re-calibrating an analytical device, or any such action. The action level for each such process will be shown in tabular form.



Bethlehem Steel Corporation  
Burns Harbor Division

WASTE ANALYSIS PLAN FOR  
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Table 1 - Analytical Methods for Waste Analysis

ANALYSIS	WASTE	ANALYTICAL METHOD
Chromium	SPL	Method No. 6010A
Lead	SPL	Method No. 7421
Selenium	WAL	Method No. 7740
Volatile Organics (Benzene)	WAL	Method No. 8260A
Semi-Volatile Organics (Pyridine)	WAL	Method No. 8270B
Specific Conductivity	SPL	Method No. 2510B
Specific Gravity	SPL/WAL	Method No. 2710F
Total Dissolved Solids (TDS)	SPL	Method No. 160.1
Total Organic Carbon (TOC)	SPL/WAL	Method No. 9060

\* Analytical methods are described in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA SW-846, Third Edition, 1986.

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Bethlehem Steel Corporation  
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WASTE ANALYSIS PLAN FOR  
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Table 2 - Constituents and Historic Concentrations

CONSTITUENTS	WASTE	MINIMUM	MAXIMUM	AVERAGE	UNITS
Chromium	SPL	59.8	89.7	73.5	mg/L
Lead	SPL	0.0	22.5	4.23	mg/L
Selenium	WAL	0.80	3.03	1.58	mg/L
Benzene	WAL	3	21.7	12.5	mg/L
Pyridine	WAL	0.0	31.2	19.9	mg/L
Specific Conductivity	SPL	90,000	328,000	164,623	umhos
Specific Gravity	SPL/WAL	1.09/1.00	1.33/1.01	1.25/1.01	g/cc
TDS	SPL	38,710	395,100	223,295	mg/L
TOC	SPL/WAL	150/1060	150/1060	150/1060	mg/L

$$1\% = \frac{1}{100} = \frac{10000}{1000000}$$

October, 1997

Bethlehem Steel Corporation  
Burns Harbor Division

WASTE ANALYSIS PLAN FOR  
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Table 3 -  
Detection Limits (DL)/Practical Quantitation Limits (PQL)/Reporting Limits (RL)

CONSTITUENTS	WASTE	AVERAGE CONCENTRATION	DL	PQL	RL	UNITS
Chromium	SPL	73.5	0.05	0.05	0.05	mg/L
Lead	SPL	4.23	0.005	0.005	0.005	mg/L
Selenium	WAL	1.58	0.005	0.005	0.005	mg/L
Benzene	WAL	12.5	5	5	5	mg/L
Pyridine	WAL	19.9	10	10	10	mg/L
Specific Conductivity	SPL	164,623	10	10	10	umhos
Specific Gravity	SPL/WAL	1.25/1.01	NA	NA	NA	
TDS	SPL	223,295	10	25	25	mg/L
TOC	SPL/WAL	150/1060	1	1	1	mg/L

Note: Data in this table assumes no sample dilutions have been performed.

Bethlehem Steel Corporation  
Burns Harbor Division

WASTE ANALYSIS PLAN FOR  
UIC Permit Nos. IN-127-1W-0001, 0003 and 0004

Attachment 2 - Waste Ammonia Liquor Appendix IX Analysis



## American Analytical Inc.

Environmental and Industrial  
Analytical Laboratory Services

7870 Broadway  
Merrillville, IN 46410  
Tel: 219-769-8378  
Fax: 219-769-1664

October 27, 1993

*Please file*  
*LSW*

Mr. Dean McDevitt  
Bethlehem Steel Company  
P.O. Box 248  
Chesterton, Indiana 46304

A<sub>2</sub>I Numbers 936781 - 936782

Dear Mr. McDevitt:

Enclosed are the Appendix IX results for the two samples that we received from you on October 19, 1993.

All Quality Control associated with these samples will be kept on file for your convenience for five years. After such time it will be disposed if we are not otherwise notified.

It has been a pleasure serving you; and if you have any questions concerning these results please do not hesitate to contact us.

Respectfully Submitted,

AMERICAN ANALYTICAL, INC.

David W. Johnson  
Project Manager/Chemist

Enclosures

DWJ:ces





# American Analytical Inc.

Environmental and Industrial  
Analytical Laboratory Services

7870 Broadway  
Merrillville, IN 46410  
Tel: 219-769-8378  
Fax: 219-769-1664

## APPENDIX IX METALS COMPOUNDS ANALYTICAL RESULTS

Client: Bethlehem Steel Corporation

Sample Identification: WAL  
Sample Matrix: Aqueous  
Date Sampled: 10/19/93  
Date Received: 10/19/93  
Date Digested: 10/22/93  
Date Analyzed: 10/25 - 10/26/93  
Analyst: MZ  
Client Project ID: N/A  
Lab Number: 936781

ANALYTE	CONCENTRATION (mg/L)	METHOD EPA 600/4-79-020	QUANTITATION LIMIT (mg/L)
Arsenic	<.100	206.2	.100
Cadmium	<.100	213.1	.100
Chromium	<.500	218.1	.500
Lead	<.500	239.1	.500
Mercury	<.010	245.1	.010
Selenium	6.76 > 1.0	270.2	1.00
Copper	<.200	220.1	.200
Nickel	<.500	249.1	.500
Thallium	<.050	279.2	.050
Zinc	.420	289.1	.200
Beryllium	<.100	210.1	.100
Antimony	<.500	204.2	.500
Barium	<2.00	208.1	2.00
Vanadium	<.200	6010	.200

Respectfully Submitted,  
American Analytical, Inc.



# American Analytical Inc.

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Analytical Laboratory Services

7870 Broadway  
Merrillville, IN 46410  
Tel: 219-769-8378  
Fax: 219-769-1664

## WET CHEMISTRY ANALYTICAL RESULTS

Client: Bethlehem Steel Corporation

Sample Identification: WAL  
Sample Matrix: Aqueous  
Date Sampled: 10/19/93  
Date Received: 10/19/93  
Date Analyzed: 10/26/93  
Analyst: SA/CB  
Client Project ID: N/A  
Lab Number: 936781

ANALYTE	CONCENTRATION (mg/L)	METHOD EPA 600/4-79-020	QUANTITATION LIMIT (mg/L)
Cyanide	<.010	335.2	.010
Sulfide	72.0	376.2	.050

Respectfully Submitted,  
American Analytical, Inc.



# American Analytical Inc.

Environmental and Industrial  
Analytical Laboratory Services

7870 Broadway  
Merrillville, IN 46410  
Tel: 219-769-8378  
Fax: 219-769-1664

PRIORITY POLLUTANTS  
PCBS  
ANALYTICAL RESULTS

Client: Bethlehem Steel Corporation

Sample Identification: WAL  
Sample Matrix: Aqueous  
Date Sampled: 10/19/93  
Date Received: 10/19/93  
Date Extracted: 10/25/93  
Date Analyzed: 10/25/93  
Analyst: TC  
Client Project ID: N/A  
Lab Number: 936781

ANALYTE	CAS NUMBER	CONCENTRATION (ug/L)	QUANTITATION LIMIT (ug/L)
PCB-1016	12974-11-2	ND	10
B-1221	11104-28-2	ND	10
PCB-1232	11141-16-5	ND	10
PCB-1242	53469-21-9	ND	10
PCB-1248	12672-29-6	ND	10
PCB-1254	11097-69-1	ND	10
PCB-1260	11096-82-5	ND	10

J - Analyte detected at levels below Quantitation Limit  
ND - Not Detected at Quantitation Limit  
Analyte Method: SW-846 8080 / EPA 603

Respectfully Submitted,  
American Analytical, Inc.

APPENDIX IX  
VOLATILE COMPOUNDS  
ANALYTICAL RESULTS

Lab Number:

Blk102693

Page 2 of 2

ANALYTE	CAS NUMBER	CONCENTRATION (ug/L)	QUANTITATION LIMIT (ug/L)
1,1,2-Trichloroethane	79-00-5	ND	5
Trichloroethene	79-01-6	ND	5
Vinyl acetate	108-05-4	ND	10
Vinyl chloride	75-01-4	ND	10
Xylenes (Total)	1330-20-7	ND	5
Acetonitrile	2206-26-0	ND	100
1,2-Dibromo-3-chloropropane	96-2-8	ND	10
1,2-Dibromoethane	106-93-4	ND	10
Dibromomethane	74-95-3	ND	10
Dichlorodifluoromethane	75-71-8	ND	10
Ethylmethacrylate	97-63-2	ND	50
Iodomethane	74-83-4	ND	100
Isobutylalcohol	78-83-1	ND	100
Methacrylonitrile	126-98-7	ND	50
Methylmethacrylate	80-62-6	ND	50
1,1,2-Tetrachloroethane	630-20-6	ND	10
chloromonofluoromethane	75-69-4	ND	10
1,2,3-Trichloropropane	96-18-4	ND	10

ND - Not Detected at Quantitation Limit

J - Analyte detected at levels below Quantitation Limit

Analyte Method: U.S. EPA 624/SW-846, 8260

Respectfully Submitted,  
American Analytical, Inc.



# American Analytical Inc.

Environmental and Industrial  
Analytical Laboratory Services

7870 Broadway  
Merrillville, IN 46410  
Tel: 219-769-8378  
Fax: 219-769-1664

## APPENDIX IX VOLATILE COMPOUNDS ANALYTICAL RESULTS

Client: Bethlehem Steel Corporation

Sample Identification: WAL  
Sample Matrix: Aqueous  
Date Sampled: 10/19/93  
Date Received: 10/19/93  
Date Analyzed: 10/26/93  
Analyst: TC  
Project Number: N/A  
Lab Number: 936781

Page 1 of 2

ANALYTE	CAS NUMBER	CONCENTRATION (ug/L)	QUANTITATION LIMIT (ug/L)
Acetone	67-64-1	ND	500
Acrolein	107-02-8	ND	500
Acrylonitrile	107-13-1	ND	500
Benzene	71-43-2	7,200 ✓ = 72 mg/L	250
Bromodichloromethane	75-27-4	ND	250
Bromoform	75-25-2	ND	250
Bromonethane	74-83-9	ND	500
2-Butanone	78-93-3	ND	500
Carbon disulfide	75-15-0	ND	250
Carbon tetrachloride	56-23-5	ND	250
Chlorobenzene	108-90-7	ND	250
Chloroethane	75-00-3	ND	500
Chloroform	67-66-3	ND	250
Chloromethane	74-87-3	ND	500
Dibromochloromethane	124-48-1	ND	250
1,1-Dichloroethane	75-34-3	ND	250
1,2-Dichloroethane	107-06-2	ND	250
1,1-Dichloroethene	75-35-4	ND	250
Trans/Cis-1,2-Dichloroethene	540-59-0	ND	250
1,2-Dichloropropane	78-87-5	ND	250
cis-1,3-Dichloropropene	10061-1-5	ND	250
trans-1,3-Dichloropropene	10061-02-5	ND	250
Ethyl benzene	100-41-4	ND	250
2-Hexanone	591-78-6	ND	500
Methylene chloride	75-09-2	ND	250
4-Methyl-2-Pentanone	108-10-1	ND	500
Styrene	100-42-5	700	250
1,1,2,2-Tetrachloroethane	79-34-5	ND	250
Tetrachloroethene	127-18-4	ND	250
Toluene	108-88-3	1,800	250
1,1,1-Trichloroethane	71-55-6	ND	250

APPENDIX IX  
VOLATILE COMPOUNDS  
ANALYTICAL RESULTS

Lab Number: 936781

Page 2 of 2

ANALYTE	CAS NUMBER	CONCENTRATION (ug/L)	QUANTITATION LIMIT (ug/L)
1,1,2-Trichloroethane	79-00-5	ND	250
Trichloroethene	79-01-6	ND	250
Vinyl acetate	108-05-4	ND	500
Vinyl chloride	75-01-4	ND	500
Xylenes (Total)	1330-20-7	620	250
Acetonitrile	2206-26-0	ND	5,000
1,2-Dibromo-3-chloropropane	96-2-8	ND	500
1,2-Dibromoethane	106-93-4	ND	500
Dibromomethane	74-95-3	ND	500
Dichlorodifluoromethane	75-71-8	ND	500
Ethylmethacrylate	97-63-2	ND	2,500
Iodomethane	74-88-4	ND	5,000
Isobutylalcohol	78-83-1	ND	5,000
Methacrylonitrile	126-98-7	ND	2,500
Methylmethacrylate	80-62-6	ND	2,500
1,1,1,2-Tetrachloroethane	630-20-6	ND	500
Chloromonoisobutylmethane	75-69-4	ND	500
1,2,3-Trichloropropane	96-18-4	ND	500

ND - Not Detected at Quantitation Limit

I - Analyte detected at levels below Quantitation Limit

Analyte Method: U.S. EPA 624/SW-846, 8260

Respectfully Submitted,  
American Analytical, Inc.



APPENDIX IX  
SEMI-VOLATILE ORGANIC COMPOUNDS  
ANALYTICAL RESULTS

Lab Number:

Blk102593

Page 2 of 4

ANALYTE	CAS NUMBER	CONCENTRATION (mg/L)	QUANTITATION LIMIT (mg/L)
2-Nitroaniline	88-74-4	ND	0.050
Dimethylphthalate	131-11-3	ND	0.010
Acenaphthylene	208-96-8	ND	0.010
2,6-Dinitrotoluene	606-20-2	ND	0.010
3-Nitroaniline	99-09-2	ND	0.050
Acenaphthene	83-32-9	ND	0.010
2,4-Dinitrophenol	51-28-5	ND	0.050
4-Nitrophenol	100-02-7	ND	0.050
Dibenzofuran	132-64-9	ND	0.010
2,4-Dinitrotoluene	121-14-2	ND	0.010
4-Chlorophenyl-phenyl ether	7005-72-3	ND	0.010
Fluorene	86-73-7	ND	0.010
4-Nitroaniline	100-01-6	ND	0.050
4,6-Dinitro-2-Methylphenol	534-52-1	ND	0.050
N-nitrosodiphenylamine	86-30-6	ND	0.010
4-Bromophenyl-phenylether	101-55-3	ND	0.010
1,2-Dichlorobenzene	118-74-1	ND	0.010
1,3-Dichlorophenol	87-86-5	ND	0.050
Phenanthrene	85-01-8	ND	0.010
Anthracene	120-12-7	ND	0.010
Di-n-butylphthalate	84-74-2	ND	0.010
Fluoranthene	206-44-0	ND	0.010
Pyrene	129-00-0	ND	0.010
Butylbenzylphthalate	85-68-7	ND	0.010
3,3'-Dichlorobenzidine	91-94-1	ND	0.050
Benzo(a)anthracene	56-55-3	ND	0.010
Chrysene	218-01-9	ND	0.010
Bis (2-ethylhexyl) phthalate	117-81-7	ND	0.010
Di-n-octylphthalate	117-84-0	ND	0.010
Benzo(b)fluoranthene	205-99-2	ND	0.010
2,4,5,6-Tetrachlorophenol	58-90-2	ND	0.050
Benzidine	92-87-5	ND	0.050
1,2-Diphenylhydrazine	122-66-7		0.010
n-Nitrosodimethylamine	67-75-9	ND	0.010
Toluenediamine	95-80-7	ND	0.050
Benzo(k)fluoranthene	207-08-9	ND	0.010
Benzo(A)pyrene	50-32-8	ND	0.010
Indeno(1,2,3-cd) pyrene	193-39-5	ND	0.010
Dibenzo(a,h)anthracene	53-70-3	ND	0.010
Benzo(g,h,i)perylene	191-24-2	ND	0.010
Diethylphthalate	84-66-2	ND	0.010
Pyridine	75-01-4	ND	0.010

APPENDIX IX  
SEMI-VOLATILE ORGANIC COMPOUNDS  
ANALYTICAL RESULTS

Lab Number:

Blk102593

Page 3 of 4

ANALYTE	CAS NUMBER	CONCENTRATION (mg/L)	QUANTITATION LIMIT (mg/L)
Acetophenone	23077-64-7	ND	0.050
2-Acetylaminofluorene	53-96-3	ND	0.050
4-Aminobiphenyl	92-67-1	ND	0.010
Aniline	62-53-3	ND	0.010
Aramite	140-57-8	ND	0.050
2-Sec-butyl-4,6-dinitrophenol	88-85-7	ND	0.050
Chlorobenzilate	510-15-6	ND	0.050
3-Methylphenol	108-39-4	ND	0.010
2-Chloro-1,3-butadiene	126-99-8	ND	0.010
Diallate	2303-16-4	ND	0.050
2,6-Dichlorophenol	87-65-0	ND	0.050
Dimethoate	60-51-5	ND	0.050
p-Dimethylaminoazobenzene	60-11-7	ND	0.050
7,12-Dimethylbenz(a)anthracene	57-97-6	ND	0.010
3,3'-Dimethylbenzidine	612-82-8	ND	0.050
Alpha, alpha-dimethylphenethylamine		ND	0.050
m-Dinitrobenzene	99-65-0	ND	0.050
p-Toluenylamine	122-39-4	ND	0.050
Disulfoton	298-04-4	ND	0.10
Ethylmethanesulfonate	62-50-0	ND	0.050
Famphur	52-85-7	ND	0.10
Hexachlorophene	70-30-4	ND	0.050
Hexachloropropene	1888-71-7	ND	0.050
Isodrin	465-73-6	ND	0.050
Isosafrole	120-58-1	ND	0.050
Methapyrilene	91-80-5	ND	0.050
3-Methylchloranthrene	56-79-5	ND	0.050
Methylmethanesulfonate	66-27-3	ND	0.050
Methylparathion	298-00-0	ND	0.050
1,4-Naphthoquinone	130-15-4	ND	0.010
1-Naphthylamine	134-32-7	ND	0.010
2-Naphthylamine	91-59-8	ND	0.010
4-Nitroquinoline 1-oxide	56-57-5	ND	0.050
N-nitrosodi-n-butylamine		ND	0.010
N-nitrosodiethylamine		ND	0.010
N-nitrosomethylamine		ND	0.010
N-nitrosomorpholine		ND	0.050
N-nitrosopiperidine		ND	0.010
N-nitrosopyrrolidine	930-55-2	ND	0.050
4-Nitro-o-toluidine		ND	0.050
Pentachlorobenzene	608-93-5	ND	0.010

APPENDIX IX  
SEMI-VOLATILE ORGANIC COMPOUNDS  
ANALYTICAL RESULTS

Lab Number:

Bik102593

Page 4 of 4

ANALYTE	CAS NUMBER	CONCENTRATION (mg/L)	QUANTITATION LIMIT (mg/L)
Pentachloroethane	76-01-7	ND	0.010
Pentachloronitrobenzene	82-68-8	ND	0.010
Phenacetin	62-44-2	ND	0.050
P-phenylenediamine	95-54-5	ND	0.010
Phorate	298-02-2	ND	0.050
Z-Picoline	109-06-8	ND	0.010
Pronamide	23950-58-5	ND	0.050
Safrole	94-59-7	ND	0.010
1,2,4,5-Tetrachlorobenzene	634-66-2	ND	0.010
2,3,4,6-Tetrachlorophenol	58-90-2	ND	0.050
O-Toluidene	95-53-4	ND	0.050
0,0,0-Triethyl phosphorothioate	126-68-1	ND	0.10

\* Not detected at quantitation limit

J - Analyte detected at levels below Quantitation Limit

Analyte Method: U.S. EPA 625

Respectfully Submitted,  
American Analytical, Inc.



A handwritten signature in black ink, appearing to be 'C. W. G.', is written over a horizontal line.



## Environmental Laboratory Services

250 West 84th Drive • Merrillville, IN 46410 • Tel: 219-769-8378 • Fax: 219-769-1664

### APPENDIX IX SEMI-VOLATILE ORGANIC COMPOUNDS ANALYTICAL RESULTS

Client: Bethlehem Steel Corporation

Sample Identification: WAL  
Sample Matrix: Aqueous  
Date Sampled: 10/19/93  
Date Received: 10/19/93  
Date Extracted: 10/25/93  
Date Analyzed: 10/26/93  
Analyst: DWJ  
Project Identification: N/A  
Lab Number: 936781

Page 1 of 4

ANALYTE	CAS NUMBER	CONCENTRATION (mg/L)	QUANTITATION LIMIT (mg/L)
Phenol	108-95-2	150	10
Bis (2-Chloroethyl) Ether	111-44-4	ND	10
2-Chlorophenol	95-57-8	ND	10
1,3-Dichlorobenzene	541-73-1	ND	10
Dichlorobenzene	106-46-7	ND	10
Benzyl Alcohol	100-51-6	ND	20
1,2-Dichlorobenzene	95-50-1	ND	10
2-Methylphenol	95-48-7	17	10
Bis (2-Chloroisopropyl) Ether	108-60-1	ND	10
4-Methylphenol	106-44-5	64	10
N-Nitrosodipropylamine	621-64-7	ND	10
Hexachloroethane	67-72-1	ND	10
Nitrobenzene	98-95-3	ND	10
Isophorone	78-59-1	ND	10
2-Nitrophenol	88-75-5	ND	10
2,4-Dimethylphenol	105-67-9	ND	10
Benzoic Acid	65-85-0	ND	50
Bis (2-Chloroethoxy) Methane	111-91-1	ND	10
2,4-Dichlorophenol	120-83-2	ND	10
1,2,4-Trichlorobenzene	120-82-1	ND	10
Naphthalene	91-20-3	11	10
4-Chloroaniline	106-47-8	ND	20
Hexachlorobutadiene	87-68-3	ND	10
4-Chloro-3-Methylphenol (para-chloro-meta-cresol)	59-50-7	ND	20
2-Methylnaphthalene	91-57-6	ND	10
Hexachlorocyclo-Pentadiene	77-47-4	ND	10
2,4,6-Trichlorophenol	88-06-2	ND	10
2,4,5-Trichlorophenol	95-95-4	ND	50
1-Chloronaphthalene	91-58-7	ND	10

APPENDIX IX  
SEMI-VOLATILE ORGANIC COMPOUNDS  
ANALYTICAL RESULTS

Lab Number:

936781

Page 2 of 4

ANALYTE	CAS NUMBER	CONCENTRATION (mg/L)	QUANTITATION LIMIT (mg/L)
2-Nitroaniline	88-74-4	ND	50
Dimethylphthalate	131-11-3	ND	10
Acenaphthylene	208-96-8	ND	10
2,6-Dinitrotoluene	606-20-2	ND	10
3-Nitroaniline	99-09-2	ND	50
Acenaphthene	83-32-9	ND	10
2,4-Dinitrophenol	51-28-5	ND	50
4-Nitrophenol	100-02-7	ND	50
Dibenzofuran	132-64-9	ND	10
2,4-Dinitrotoluene	121-14-2	ND	10
4-Chlorophenyl-phenyl ether	7005-72-3	ND	10
Fluorene	86-73-7	ND	10
4-Nitroaniline	100-01-6	ND	50
4,6-Dinitro-2-Methylphenol	534-52-1	ND	50
N-nitrosodiphenylamine	86-30-6	ND	10
4-Bromophenyl-phenylether	101-55-3	ND	10
Hexachlorobenzene	118-74-1	ND	10
2,4-Dichlorophenol	87-86-5	ND	50
Fluoranthrene	85-01-8	ND	10
Anthracene	120-12-7	ND	10
Di-n-butylphthalate	84-74-2	ND	10
Fluoranthene	206-44-0	ND	10
Pyrene	129-00-0	ND	10
Butylbenzylphthalate	85-68-7	ND	10
3,3'-Dichlorobenzidine	91-94-1	ND	50
Benzo(a)anthracene	56-55-3	ND	10
Chrysene	218-01-9	ND	10
Bis (2-ethylhexyl) phthalate	117-81-7	ND	10
Di-n-octylphthalate	117-84-0	ND	10
Benzo(b)fluoranthene	205-99-2	ND	10
2,4,5,6-Tetrachlorophenol	58-90-2	ND	50
Benzidine	92-87-5	ND	50
1,2-Diphenylhydrazine	122-66-7	?	10
n-Nitrosodimethylamine	67-75-9	ND	10
Toluenediamine	95-80-7	ND	50
Benzo(k)fluoranthene	207-08-9	ND	10
Benzo(A)pyrene	50-32-8	ND	10
Indeno(1,2,3-cd) pyrene	193-39-5	ND	10
Dibenzo(a,h)anthracene	53-70-3	ND	10
Benzo(g,h,i)perylene	191-24-2	ND	10
Diethylphthalate	84-66-2	ND	10
Pyridine	75-01-4	11	10

APPENDIX IX  
SEMI-VOLATILE ORGANIC COMPOUNDS  
ANALYTICAL RESULTS

Lab Number: 936781

Page 3 of 4

ANALYTE	CAS NUMBER	CONCENTRATION (mg/L)	QUANTITATION LIMIT (mg/L)
Acetophenone	28077-64-7	ND	50
2-Acetylaminofluorene	53-96-3	ND	50
4-Aminobiphenyl	92-67-1	ND	10
Aniline	62-53-3	9 J	10
Aramite	140-57-8	ND	50
2-Sec-butyl-4,6-dinitrophenol	88-85-7	ND	50
Chlorobenzilate	510-15-6	ND	50
3-Methylphenol	108-39-4	ND	10
2-Chloro-1,3-butadiene	126-99-8	ND	10
Diallate	2303-16-4	ND	50
2,6-Dichlorophenol	87-65-0	ND	50
Dimethoate	60-51-5	ND	50
p-Dimethylaminoazobenzene	60-11-7	ND	50
7,12-Dimethylbenz(a)anthracene	57-97-6	ND	10
3,3'-Dimethylbenzidine	612-82-8	ND	50
Alpha, alpha-dimethylphenethylamine		ND	50
m-Dinitrobenzene	99-65-0	ND	50
henylamine	122-39-4	ND	50
Disulfoton	298-04-4	ND	100
Ethylmethanesulfonate	62-50-0	ND	50
Famphur	52-85-7	ND	100
Hexachlorophene	70-30-4	ND	50
Hexachloropropene	1888-71-7	ND	50
Isodrin	465-73-6	ND	50
Isosafrole	120-58-1	ND	50
Methapyrilene	91-80-5	ND	50
3-Methylchloranthrene	56-79-5	ND	50
Methylmethanesulfonate	66-27-3	ND	50
Methylparathion	298-00-0	ND	50
1,4-Naphthoquinone	130-15-4	ND	10
1-Naphthylamine	134-32-7	ND	10
2-Naphthylamine	91-59-8	ND	10
4-Nitroquinoline 1-oxide	56-57-5	ND	50
N-nitrosodi-n-butylamine		ND	10
N-nitrosodiethylamine		ND	10
N-nitrosomethylamine		ND	10
N-nitrosomorpholine		ND	50
N-nitrosopiperidine		ND	10
N-nitrosopyrrolidine	930-55-2	ND	50
5-Nitro-o-toluidine		ND	50
Pentachlorobenzene	608-93-5	ND	10

APPENDIX IX  
SEMI-VOLATILE ORGANIC COMPOUNDS  
ANALYTICAL RESULTS

Lab Number: 936781

Page 4 of 4

ANALYTE	CAS NUMBER	CONCENTRATION (mg/L)	QUANTITATION LIMIT (mg/L)
Pentachloroethane	76-01-7	ND	10
Pentachloronitrobenzene	82-68-8	ND	10
Phenacetin	62-44-2	ND	50
P-phenylenediamine	95-54-5	ND	10
Phorate	298-02-2	ND	50
Z-Picoline	109-06-8	ND	10
Pronamide	23950-58-5	ND	50
Safrole	94-59-7	ND	10
1,2,4,5-Tetrachlorobenzene	634-66-2	ND	10
2,3,4,6-Tetrachlorophenol	58-90-2	ND	50
O-Toluidene	95-53-4	ND	50
0,0,0-Triethyl phosphorothioate	126-68-1	ND	100

I - Not detected at quantitation limit

J - Analyte detected at levels below Quantitation Limit

Analyte Method: U.S. EPA 625

Respectfully Submitted,  
American Analytical, Inc.





Bethlehem Steel Corporation  
Burns Harbor Division

WASTE ANALYSIS PLAN FOR  
UIC Permit Nos. IN-127-1W-0001, 0003 and 0004

*pinkle*  
Attachment 2 - Waste Ammonia Liquor Appendix IX Analysis



# American Analytical Inc.

Environmental and Industrial  
Analytical Laboratory Services

7870 Broadway  
Merrillville, IN 46410  
Tel: 219-769-8378  
Fax: 219-769-1664

October 27, 1993

*Phase file*  
*LSW*

Mr. Dean McDevitt  
Bethlehem Steel Company  
P.O. Box 248  
Chesterton, Indiana 46304

A<sub>2</sub>I Numbers 936781 - 936782

Dear Mr. McDevitt:

Enclosed are the Appendix IX results for the two samples that we received from you on October 19, 1993.

All Quality Control associated with these samples will be kept on file for your convenience for five years. After such time it will be disposed if we are not otherwise notified.

It has been a pleasure serving you; and if you have any questions concerning these results please do not hesitate to contact us.

Respectfully Submitted,

AMERICAN ANALYTICAL, INC.

David W. Johnson  
Project Manager/Chemist

Enclosures

DWJ:ces





# American Analytical Inc.

Environmental and Industrial  
Analytical Laboratory Services

7870 Broadway  
Merrillville, IN 46410  
Tel: 219-769-8378  
Fax: 219-769-1664

## APPENDIX IX METALS COMPOUNDS ANALYTICAL RESULTS

Client: Bethlehem Steel Corporation

Sample Identification: WPL

Sample Matrix: Aqueous

Date Sampled: 10/19/93

Date Received: 10/19/93

Date Digested: 10/22/93

Date Analyzed: 10/25 - 10/26/93

Analyst: MZ

Client Project ID: N/A

Lab Number: 936782

ANALYTE	CONCENTRATION (mg/L)	METHOD EPA 600/4-79-020	QUANTITATION LIMIT (mg/L)
Arsenic	.238 < 5.0	206.2	.100
Cadmium	<.100	213.1	.100
Chromium	60.1 > 5.0	218.1	.500
Lead	<.500	239.1	.500
Mercury	<.010	245.1	.010
Selenium	<.050	270.2	1.00
Copper	11.4	220.1	.200
Nickel	16.5 0.44 mg/L	249.1	.500
Thallium	<.050	279.2	.050
Zinc	2.11	289.1	.200
Beryllium	<.100	210.1	.100
Antimony	<.500	204.2	.500
Barium	<2.00	208.1	2.00
Vanadium	7.70	6010	.200

Respectfully Submitted,  
American Analytical, Inc.



# American Analytical Inc.

Environmental and Industrial  
Analytical Laboratory Services

7870 Broadway  
Merrillville, IN 46410  
Tel: 219-769-8378  
Fax: 219-769-1664

## WET CHEMISTRY ANALYTICAL RESULTS

Client: Bethlehem Steel Corporation

Sample Identification: WPL  
Sample Matrix: Aqueous  
Date Sampled: 10/19/93  
Date Received: 10/19/93  
Date Analyzed: 10/26/93  
Analyst: SA/CB  
Client Project ID: N/A  
Lab Number: 936782

ANALYTE	CONCENTRATION (mg/L)	METHOD EPA 600/4-79-020	QUANTITATION LIMIT (mg/L)
Cyanide	<.010	335.2	.010
Sulfide	<.050	376.2	.050

Respectfully Submitted,  
American Analytical, Inc.



# American Analytical Inc.

Environmental and Industrial  
Analytical Laboratory Services

7870 Broadway  
Merrillville, IN 46410  
Tel: 219-769-8378  
Fax: 219-769-1664

PRIORITY POLLUTANTS  
PCBS  
ANALYTICAL RESULTS

Client: Bethlehem Steel Corporation

Sample Identification: WPL  
Sample Matrix: Aqueous  
Date Sampled: 10/19/93  
Date Received: 10/19/93  
Date Extracted: 10/25/93  
Date Analyzed: 10/25/93  
Analyst: TC  
Client Project ID: N/A  
Lab Number: 936782

ANALYTE	CAS NUMBER	CONCENTRATION (ug/L)	QUANTITATION LIMIT (ug/L)
PCB-1016	12974-11-2	ND	10
PCB-1221	11104-28-2	ND	10
PCB-1232	11141-16-5	ND	10
PCB-1242	53469-21-9	ND	10
PCB-1243	12672-29-6	ND	10
PCB-1254	11097-69-1	ND	10
PCB-1260	11096-82-5	ND	10

J - Analyte detected at levels below Quantitation Limit

ND - Not Detected at Quantitation Limit

Analyte Method: SW-846 8080 / EPA 608

Respectfully Submitted,  
American Analytical, Inc.



# American Analytical Inc.

Environmental and Industrial  
Analytical Laboratory Services

7870 Broadway  
Merrillville, IN 46410  
Tel: 219-769-8378  
Fax: 219-769-1664

## APPENDIX IX VOLATILE COMPOUNDS ANALYTICAL RESULTS

Client: Bethlehem Steel Corporation

Sample Identification: WPL  
Sample Matrix: Aqueous  
Date Sampled: 10/19/93  
Date Received: 10/19/93  
Date Analyzed: 10/26/93  
Analyst: TC  
Project Number: N/A  
Lab Number: 936782

Page 1 of 2

ANALYTE	CAS NUMBER	CONCENTRATION (ug/L)	QUANTITATION LIMIT (ug/L)
Acetone	67-64-1	ND	120
Acrolein	107-02-8	ND	120
Acrylonitrile	107-13-1	ND	120
Benzene	71-43-2	ND	62
Bromodichloromethane	75-27-4	ND	62
Bromoform	75-25-2	ND	62
Bromomethane	74-83-9	ND	120
2-Butanone	78-93-3	ND	120
Carbon disulfide	75-15-0	ND	62
Carbon tetrachloride	56-23-5	ND	62
Chlorobenzene	108-90-7	ND	62
Chloroethane	75-00-3	ND	120
Chloroform	67-66-3	ND	62
Chloromethane	74-87-3	ND	120
Dibromochloromethane	124-48-1	ND	62
1,1-Dichloroethane	75-34-3	ND	62
1,2-Dichloroethane	107-06-2	1,100 > 500	62
1,1-Dichloroethene	75-35-4	ND	62
Trans/Cis-1,2-Dichloroethene	540-59-0	ND	62
1,2-Dichloropropane	78-87-5	ND	62
cis-1,3-Dichloropropene	10061-1-5	ND	62
trans-1,3-Dichloropropene	10061-02-5	ND	62
Ethyl benzene	100-41-4	ND	62
2-Hexanone	591-78-6	ND	120
Methylene chloride	75-09-2	ND	62
4-Methyl-2-Pentanone	108-10-1	ND	120
Styrene	100-42-5	ND	62
1,1,2,2-Tetrachloroethane	79-34-5	ND	62
Tetrachloroethene	127-18-4	ND	62
Toluene	108-88-3	ND	62
1-Trichloroethane	71-55-6	ND	62

APPENDIX IX  
VOLATILE COMPOUNDS  
ANALYTICAL RESULTS

Lab Number: 936782

Page 2 of 2

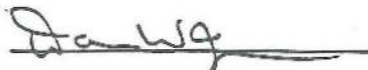
ANALYTE	CAS NUMBER	CONCENTRATION	QUANTITATION
		(ug/L)	LIMIT (ug/L)
1,1,2-Trichloroethane	79-00-5	ND	62
Trichloroethene	79-01-6	ND	62
Vinyl acetate	108-05-4	ND	120
Vinyl chloride	75-01-4	ND	120
Xylenes (Total)	1330-20-7	ND	62
Acetonitrile	2206-26-0	ND	1,200
1,2-Dibromo-3-chloropropane	96-2-8	ND	120
1,2-Dibromoethane	106-93-4	ND	120
Dibromomethane	74-95-3	ND	120
Dichlorodifluoromethane	75-71-8	ND	120
Ethylmethacrylate	97-63-2	ND	600
Iodomethane	74-88-4	ND	1,200
Isobutylalcohol	78-83-1	ND	1,200
Methacrylonitrile	126-98-7	ND	600
Methylmethacrylate	80-62-6	ND	600
1,1,1,2-Tetrachloroethane	630-20-6	ND	120
Trichloromonofluoromethane	75-69-4	ND	120
1,2,3-Trichloropropane	96-18-4	ND	120

ND - Not Detected at Quantitation Limit

J - Analyte detected at levels below Quantitation Limit

Analyte Method: U.S. EPA 624/SW-846, 8260

Respectfully Submitted,  
American Analytical, Inc.





# American Analytical Inc.

Environmental and Industrial  
Analytical Laboratory Services

7870 Broadway  
Merrillville, IN 46410  
Tel: 219-769-8378  
Fax: 219-769-1664

## APPENDIX IX SEMI-VOLATILE ORGANIC COMPOUNDS ANALYTICAL RESULTS

Client: Bethlehem Steel Corporation

Sample Identification: WPL  
Sample Matrix: Aqueous  
Date Sampled: 10/19/93  
Date Received: 10/19/93  
Date Extracted: 10/25/93  
Date Analyzed: 10/25/93  
Analyst: DWJ  
Project Identification: N/A  
Lab Number: 936782

Page 1 of 4

ANALYTE	CAS NUMBER	CONCENTRATION (mg/L)	QUANTITATION LIMIT (mg/L)
Phenol	108-95-2	ND	0.10
Bis (2-Chloroethyl) Ether	111-44-4	ND	0.10
2-Chlorophenol	95-57-8	ND	0.10
1,3-Dichlorobenzene	541-73-1	ND	0.10
1,4-Dichlorobenzene	106-46-7	ND	0.10
Benzyl Alcohol	100-51-6	ND	0.20
1,2-Dichlorobenzene	95-50-1	ND	0.10
2-Methylphenol	95-48-7	ND	0.10
Bis (2-Chloroisopropyl) Ether	108-60-1	ND	0.10
4-Methylphenol	106-44-5	ND	0.10
N-Nitrosodipropylamine	621-64-7	ND	0.10
Hexachloroethane	67-72-1	ND	0.10
Nitrobenzene	98-95-3	ND	0.10
Isophorone	78-59-1	ND	0.10
2-Nitrophenol	88-75-5	ND	0.10
2,4-Dimethylphenol	105-67-9	ND	0.10
Benzoic Acid	65-85-0	ND	0.50
Bis (2-Chloroethoxy) Methane	111-91-1	ND	0.10
2,4-Dichlorophenol	120-83-2	ND	0.10
1,2,4-Trichlorobenzene	120-82-1	ND	0.10
Naphthalene	91-20-3	ND	0.10
4-Chloroaniline	106-47-8	ND	0.20
Hexachlorobutadiene	87-68-3	ND	0.10
4-Chloro-3-Methylphenol (para-chloro-meta-cresol)	59-50-7	ND	0.20
2-Methylnaphthalene	91-57-6	ND	0.10
Hexachlorocyclo-Pentadiene	77-47-4	ND	0.10
2,4,6-Trichlorophenol	88-06-2	ND	0.10
2,4,5-Trichlorophenol	95-95-4	ND	0.50
2-Chloronaphthalene	91-58-7	ND	0.10

APPENDIX IX  
SEMI-VOLATILE ORGANIC COMPOUNDS  
ANALYTICAL RESULTS

Lab Number: 936782

Page 2 of 4

ANALYTE	CAS NUMBER	CONCENTRATION (mg/L)	QUANTITATION LIMIT (mg/L)
2-Nitroaniline	88-74-4	ND	0.50
Dimethylphthalate	131-11-3	ND	0.10
Accenaphthylene	208-96-8	ND	0.10
2,6-Dinitrotoluene	606-20-2	ND	0.10
3-Nitroaniline	99-09-2	ND	0.50
Acenaphthene	83-32-9	ND	0.10
2,4-Dinitrophenol	51-28-5	ND	0.50
4-Nitrophenol	100-02-7	ND	0.50
Dibenzofuran	132-64-9	ND	0.10
2,4-Dinitrotoluene	121-14-2	ND	0.10
4-Chlorophenyy-phenyl ether	7005-72-3	ND	0.10
Fluorene	86-73-7	ND	0.10
4-Nitroaniline	100-01-6	ND	0.50
4,6-Dinitro-2-Methylphenol	534-52-1	ND	0.50
N-nitrosodiphenylamine	86-30-6	ND	0.10
4-Bromophenyl-phenlether	101-55-3	ND	0.10
Hexachlorobenzene	118-74-1	ND	0.10
Pentachlorophenol	87-86-5	ND	0.50
Phenanthrene	85-01-8	ND	0.10
Anthracene	120-12-7	ND	0.10
Di-n-butylphthalate	84-74-2	ND	0.10
Fluoranthene	206-44-0	ND	0.10
Pyrene	129-00-0	ND	0.10
Butylbenzylphthalate	85-68-7	ND	0.10
3,3'Dichlorobenzidine	91-94-1	ND	0.50
Benzo(a)anthracene	56-55-3	ND	0.10
Chrysene	218-01-9	ND	0.10
Bis (2-ethylhexyl) phthalate	117-81-7	ND	0.10
Di-n-octylphthalate	117-84-0	ND	0.10
Benzo(b)fluoranthene	205-99-2	ND	0.10
2,4,5,6-Tetrachlorophenol	58-90-2	ND	0.50
Benzidine	92-87-5	ND	0.50
1,2-Diphenylhydrazine	122-66-7		0.10
n-Nitrosodimethylamine	67-75-9	ND	0.10
Toluenediamine	95-80-7	ND	0.50
Benzo(k)fluoranthene	207-08-9	ND	0.10
Benzo(A)pyrene	50-32-8	ND	0.10
Indeno(1,2,3-cd) pyrene	193-39-5	ND	0.10
Dibenzo(a,h)anthracene	53-70-3	ND	0.10
Benzo(g,h,i)perylene	191-24-2	ND	0.10
Diethylphthalate	84-66-2	ND	0.10
Pyridine	75-01-4	ND	0.10

APPENDIX IX  
SEMI-VOLATILE ORGANIC COMPOUNDS  
ANALYTICAL RESULTS

Lab Number: 936782

Page 3 of 4

ANALYTE	CAS NUMBER	CONCENTRATION (mg/L)	QUANTITATION LIMIT (mg/L)
Acetophenone	28077-64-7	ND	0.50
2-Acetylaminofluorene	53-96-3	ND	0.50
4-Aminobiphenyl	92-67-1	ND	0.10
Aniline	62-53-3	ND	0.10
Aramite	140-57-8	ND	0.50
2-Sec-butyl-4,6-dinitrophenol	88-85-7	ND	0.50
Chlorobenzilate	510-15-6	ND	0.50
3-Methylphenol	108-39-4	ND	0.10
2-Chloro-1,3-butadiene	126-99-8	ND	0.10
Diallate	2303-16-4	ND	0.50
2,6-Dichlorophenol	87-65-0	ND	0.50
Dimethoate	60-51-5	ND	0.50
p-Dimethylaminoazobenzene	60-11-7	ND	0.50
7,12-Dimethylbenz(a)anthracene	57-97-6	ND	0.10
3,3'-Dimethylbenzidine	612-82-8	ND	0.50
Alpha, alpha-dimethylphenethylamine		ND	0.50
m-Dinitrobenzene	99-65-0	ND	0.50
iphenylamine	122-39-4	ND	0.50
Disulfoton	298-04-4	ND	1.0
Ethylmethanesulfonate	62-50-0	ND	0.50
Famphur	52-85-7	ND	1.0
Hexachlorophene	70-30-4	ND	0.50
Hexachloropropene	1888-71-7	ND	0.50
Isodrin	465-73-6	ND	0.50
Isosafrole	120-58-1	ND	0.50
Methapyrilene	91-80-5	ND	0.50
3-Methylchloranthrene	56-79-5	ND	0.50
Methylmethanesulfonate	66-27-3	ND	0.50
Methylparathion	298-00-0	ND	0.50
1,4-Naphthoquinone	130-15-4	ND	0.10
1-Naphthylamine	134-32-7	ND	0.10
2-Naphthylamine	91-59-8	ND	0.10
4-Nitroquinoline 1-oxide	56-57-5	ND	0.50
N-nitrosodi-n-butylamine		ND	0.10
N-nitrosodiethylamine		ND	0.10
N-nitrosomethylamine		ND	0.10
N-nitrosomorpholine		ND	0.50
N-nitrosopiperidine		ND	0.10
N-nitrosopyrrolidine	930-55-2	ND	0.50
5-Nitro-o-toluidine		ND	0.50
Pentachlorobenzene	608-93-5	ND	0.10

APPENDIX IX  
SEMI-VOLATILE ORGANIC COMPOUNDS  
ANALYTICAL RESULTS

Lab Number:

936782

Page 4 of 4

ANALYTE	CAS NUMBER	CONCENTRATION (mg/L)	QUANTITATION LIMIT (mg/L)
Pentachloroethane	76-01-7	ND	0.10
Pentachloronitrobenzene	82-68-8	ND	0.10
Phenacetin	62-44-2	ND	0.50
P-phenylenediamine	95-54-5	ND	0.10
Phorate	298-02-2	ND	0.50
Z-Picoline	109-06-8	ND	0.10
Pronamide	23950-58-5	ND	0.50
Safrole	94-59-7	ND	0.10
1,2,4,5-Tetrachlorobenzene	634-66-2	ND	0.10
2,3,4,6-Tetrachlorophenol	58-90-2	ND	0.50
O-Toluidene	95-53-4	ND	0.50
0,0,0-Triethyl phosphorothioate	126-68-1	ND	1.0
Pyridine	75-01-4	ND	0.10

ND - Not detected at quantitation limit

J - Analyte detected at levels below Quantitation Limit

Analyte Method: U.S. EPA 625

Respectfully Submitted,  
American Analytical, Inc.





Bethlehem Steel Corporation  
Burns Harbor Division

WASTE ANALYSIS PLAN FOR  
UIC Permit Nos. IN-127-1W-0001, 0003 and 0004

Attachment 4 - Sample Label, Sample Seal and Chain of Custody Form



ENVIRONMENTAL SAMPLING SUPPLY

LOT #  
SAMPLE  
ID

SAMPLED BY	DATE
	TIME
LOCATION	PRESERVATIVE
ANALYSIS	CLIENT

9601 San Leandro Street, Oakland, California 94603  
(510) 562-4988 (800) 233-8425

# CUSTODY SEAL



ENVIRONMENTAL SAMPLING SUPPLY

9601 San Leandro St. Oakland, CA 94625 800-233-8425

Date: \_\_\_\_\_

Signature: \_\_\_\_\_

*Number* 19115

Page \_\_\_\_\_ of \_\_\_\_\_

Client					Project Name:					Project No.					
Address					Location:										
Telephone No.					Turnaround Time (circle one):										
					24 Hour		48Hour		72 Hour		3-5 Days		7-10 Days		
Contact person to telephone / fax (circle one) results to: _____ Fax No. _____															
Purchase Order No. (or other billing reference): _____										ANALYSIS REQUESTED					
Sampled by (PRINT):					Sampler Signature:										
Sample #	Date	Time	Sample Description	# of Containers	Matrix	Filtered (Y/N)	Preserved								Laboratory Identification
COMMENTS:					Relinquished by:			Date/Time		Received by:			Date/Time		

October, 1997

Bethlehem Steel Corporation  
Burns Harbor Division

WASTE ANALYSIS PLAN FOR  
UIC Permit Nos. IN-127-1W-0001, 0003 and 0004

Appendix 1 - Standard Operating Procedures




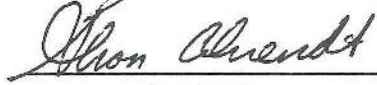
Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-6010A-1  
Rev. Number: 1.2  
Rev. Date: February 6, 1995

## Standard Operating Procedure For Inductively Coupled Plasma Methods

Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 6010A

Revision # 1.2  
Issued: February 6, 1995

	<u>9-26-96</u>
Immediate Supervisor	Date
	<u>9-26-96</u>
Second Supervisor	Date
	<u>9-26-96</u>
QA/QC Officer	Date
	<u>9-26-96</u>
Analyst	Date

Effective: February 6, 1995

### CAUTION

**Disclaimer:** This Standard Operating Procedure has been prepared for the sole use of American Analytical, Inc. and may not be specifically applicable to the activities of other organizations.

Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-6010A-1  
Rev. Number: 1.2  
Rev. Date: February 6, 1995

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SOP ID: SOP-MET-6010A-1  
Rev. Number: 1.2  
Rev. Date: February 6, 1995  
Page: 1

## **STANDARD OPERATING PROCEDURE** **INDUCTIVELY COUPLED PLASMA METHODS**

### **REFERENCE:**

SW-846, 3rd Edition, Method 6010A  
USEPA CLP SOW ILM02.0

### **MATRIX:**

Water, Leachate, Soil, Solids, Sludge

### **DETECTION LIMITS:**

For EQL's and MDL's see Table 1-1 attached.

### **RANGE:**

The effective range of this method varies per element, and is verified by quarterly linear range studies.

### **PRINCIPLE, SCOPE AND APPLICATION:**

Inductively coupled plasma emission spectroscopy is used in the determination of trace elements, including metals in solution. Prior to analysis, digestion is necessary to place the trace metal elements in solution.

Before analysis can take place, samples must be digested using appropriate preparation methods. Digestion is not required when analyzing dissolved constituents as long as samples are filter and preserved with acid.

The analysis of metals using the ICP is based on the theory of Atomic Emission. Samples are nebulized which creates an aerosol that is transported to the plasma via a torch. The plasma places the element in an atomic state which is then excited and in turn emits a light or spectra which is specific to that particular element. The spectra are separated by a grating and the intensities of the lines are measured by either photo multiplier tubes or in this case, a charged injection devise. In simpler terms, the color or wavelength of light determines which element and the intensity of the light omitted determines concentration. Background light intensity must be measured adjacent to the analyte lines during analysis. The position selected for background measurement will be determined by the complexity of the spectrum adjacent to the analytical line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not necessary when the analyte wavelength is broad and therefore may be degraded as a result of background correction. Additional interferences discussed in the next section should be appraised so that correlations can be drawn.

### INTERFERENCES AND CORRECTIVE ACTION:

Spectral interferences are caused by overlap of spectral lines from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and stray light from the line emission of high concentration elements. Spectral overlap can be compensated for by computer correction of the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line. Potential spectral interferences for recommended wavelengths have been documented but may differ for each instrument. Therefore spectral interferences must be measured for a particular instrument by aspirating 100 mg/L of the interfering element in order to measure the false analyte concentration that can arise. A correction factor can then be calculated and stored in the computer for future corrections. For example, aspirating a 100 mg/L standard of AL produces a false signal of 1.3 mg/L of Arsenic at the 193.696 line. Therefore, a correction factor can be used to compensate for this false signal.

Physical interferences are effects associated with the sample nebulization and transport processes. Samples containing high dissolved solids can cause inaccuracies. These inaccuracies can be reduced by dilution and/or a peristaltic pump.

Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects. These effects are not significant with the ICP. They can be minimized by careful selection of operating conditions, by matrix matching and by standard addition.

### SAFETY PRECAUTIONS:

Lab coats and safety glasses are to be worn at all times. All instrument vapors are to be vented to exterior of building, and all digestions are to occur under a fume hood.

### SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Aqueous and leachate samples are to be collected in 500ml plastic containers with teflon lined lids, and preserved to  $\text{pH} \leq 2$  with nitric acid. Non-aqueous samples are to be collected in 4 oz. squat jars, with teflon lined lids, and cooled to 4°C until digestion. Samples must be analyzed within 6 months of collection.

### APPARATUS:

Inductively coupled argon plasma emission spectrometer which includes a computer controlled emission spectrometer with background correction, and a radio frequency generator compliant with FCC regulations.

Liquid Argon

SOP ID: SOP-MET-6010A-1  
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Operation conditions for simple aqueous samples are as follows:

Nebulizer pressure: 33 psi  
Torch flow: High  
Auxiliary gas: Low  
RF power: 1150 W

Class A volumetric flasks

Class A volumetric pipets

#### ROUTINE MAINTENANCE:

Autosampler components, and tubing are checked daily. Tubing is changed monthly. Filters are cleaned twice yearly. Gas flow checks, and nebulizer cleaning are conducted as needed.

#### REAGENTS AND CALIBRATION STANDARDS:

Trace metal grade Hydrochloric Acid

Trace metal grade Nitric Acid

Reagent water - use Type II water for the preparation of all reagents and standards.

Standard stock solutions are purchased by supplier.

ICAP Standard Solution 1: Combine 0.10 ml A<sub>2</sub>I CAL-1(Inorganic Ventures) and 0.10 ml A<sub>2</sub>I CAL-2(Inorganic Ventures) with 2.0 ml concentrated nitric acid and 5.0 ml hydrochloric acid. Bring to final volume of 100 ml in volumetric flask.

ICAP Standard Solution 2: Combine 1.0 ml A<sub>2</sub>I CAL-1(Inorganic Ventures) and 1.0 ml A<sub>2</sub>I CAL-2(Inorganic Ventures) with 1.9 ml concentrated nitric acid, and 5.0 ml hydrochloric acid. Bring to a final volume of 100 ml in a volumetric flask.

ICAP Standard Solution 3: Combine 10 ml A<sub>2</sub>I CAL-1(Inorganic Ventures) and 10 ml A<sub>2</sub>I CAL-2(Inorganic Ventures) with 1.3 ml concentrated nitric acid, and 5.0 ml hydrochloric acid. Bring to a final volume of 100 ml in a volumetric flask.

A calibration blank is prepared by acidifying water to the same percentage of acids found in the standards and samples.

A reagent blank or preparatory blank must contain all reagents in the same volume used in the processing of the samples. This blank must be carried through the complete digestion procedure.

The instrument check standards are purchased from supplier and are prepared with the same acid concentrations as samples and standards.

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ICP ICSA Solution: Combine 10.0 ml INT-A1(Spex) with 2.0 ml concentrated nitric acid, and 5.0 ml concentrated hydrochloric acid. Bring to a final volume of 100 ml with DI water in a volumetric flask.

ICP ICSAB Solution: Combine 10.0 ml INT-A1(Spex) with 1.0 ml INT-B1(Spex), 2.0 ml concentrated nitric acid, and 5.0 ml concentrated hydrochloric acid. Bring to a final volume of 100 ml with DI water in a volumetric flask.

ICP CCV Solution: Combine 1.0 ml each of CLPP-C1CV-1, CLPP-C1CV-2, CLPP-C1CV-3, with 2.0 ml concentrated nitric acid, and 5.0 ml concentrated hydrochloric acid. Bring to a final volume of 100 ml with DI water in a volumetric flask.

ICP ICV Solution: Combine 0.20 ml each of CLPP-C1CV-1, CLPP-C1CV-2, CLPP-C1CV-3, with 2.0 ml concentrated nitric acid, and 5.0 ml concentrated hydrochloric acid. Bring to a final volume of 100 ml with DI water in a volumetric flask.

#### CALIBRATION PROCEDURES:

A four point calibration consisting of a blank and three standards is analyzed. An ICV is immediately run after the two point curve. The ICV must recover all elements between 90 - 110 % of their true value for calibration to pass. In addition, an ICB is analyzed and can not demonstrate contamination above the analyte EQL.

A CCV, followed by a CCB, is analyzed after every 10 samples. The CCV must recover within 90 - 110% of true values. The CCB must demonstrate no contamination above the EQL.

Before samples are analyzed in a run, and at the conclusion of each run, interference check samples are analyzed. The analytes of concern must recover within 20 % of their true values.

#### SAMPLE PREPARATION:

Aqueous: See SOP-MET-3005-1

Non-aqueous: See SOP-MET-3050-1

#### ANALYSIS PROCEDURE:

- 1.) Turn hoods on.
- 2.) Make sure waste bucket is not full.
- 3.) Turn on computer system, and printer.
- 4.) Turn on ICP, and autosampler.
- 5.) Click on applications manager.

- 6.) Click on analysis
- 7.) Click on CID control panel.
- 8.) Reset camera (choose hard reset)
- 9.) Set the three purge valves to 2.0 L/min.
- 10.) Place sample draw tube in DI reservoir.
- 11.) Click ignite.
- 12.) Once torch ignites, adjust sample tube clamps to get a rate of about 1.3 ml/min.
- 13.) Let torch warm up for no less than 1 hour.
- 14.) Turn on autosampler and printer.
- 15.) Click on method and specify method to be used.
- 16.) Click on autosample icon with page displayed.
- 17.) Click on samples.
- 18.) Click on add set of samples.
- 19.) Input operator I.D., A<sub>2</sub>I sample number and any other pertinent information.
- 20.) Save sample set.
- 21.) Click on table.
- 22.) Click on add a set.
- 23.) Input type, protocol, method and recall sample set, also set rinse time.
- 24.) Save table.
- 25.) Make rinse solution (1 % HNO<sub>3</sub> and 2 % HCl in DI water).
- 26.) Click on autosample icon with page displayed.
- 27.) Click on "Manual".
- 28.) Click "Move to rinse".
- 29.) Place sample draw tube in autosampler arm position over rinse reservoir.
- 30.) Set up standards and calibration checks into clean autosampler cups.

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- 31.) Pour out samples into culture tubes and put into correct order, as stated in sample table.
- 32.) Start run by clicking on auto sampler icon without page.
- 33.) Open autosample table to be run and click again.
- 34.) Set starting sample and terminating action.
- 35.) Click run.

#### QUALITY CONTROL:

Dilute and reanalyze samples that are more concentrated than the linear range or use an alternate, less sensitive line.

Analyze a reagent or prep blank for every 20 samples. Blank must fall below the EQL. If blank shows contamination a redigestion and analysis shall be performed.

Analyze replicate samples for every 20 samples to verify precision of method. If the concentration is 10 times greater than instrument detection limit, the relative percent difference (RPD) shall be within 15% for waters, and 30 % for non-aqueous samples.

Spiked replicate samples are to be analyzed every 20 samples and recovery is to be within 20% of the actual value for waters, and 30% for non-aqueous samples.

Verify calibration every 10 samples and at the end of the run using the instrument check standard and calibration blank. The check standard should agree within 10% of the actual value. The blank must be less than the EQL. If they do not, the analysis will be terminated and the problem corrected followed by calibration and reanalysis.

#### DATA TREATMENT:

For determination of metal concentration by ICP, read the metal value in mg/L from the read-out system of the instrument.

If dilution of sample was required:

$$\text{mg/L metal in sample} = A \times \frac{(C + B)}{C}$$

where,

A	=	mg/L of metal in diluted aliquot from calibration curve
B	=	Acid blank matrix used for dilution, mL
C	=	Sample aliquot, mL

For non-aqueous samples, report all concentrations as mg/Kg, hence:

$$\text{mg metal/kg sample} = \frac{A \times V}{W}$$

where,

A = mg/L of metal in processed sample from  
 instrument read out  
 V = final volume of processed sample, mL  
 W = weight of sample, g

The relative percent difference is to be calculated as follows:

$$\text{RPD} = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

RPD = Relative Percent Difference  
 D<sub>1</sub> = First Sample Value  
 D<sub>2</sub> = Second Sample Value (replicate or duplicate)

The spike recovery is to be calculated as follows:

$$\% R = \frac{SS - S}{SA} \times 100$$

% R = Percent Recovered  
 SS = Spike Sample Value  
 S = Sample Value  
 SA = Spike Added

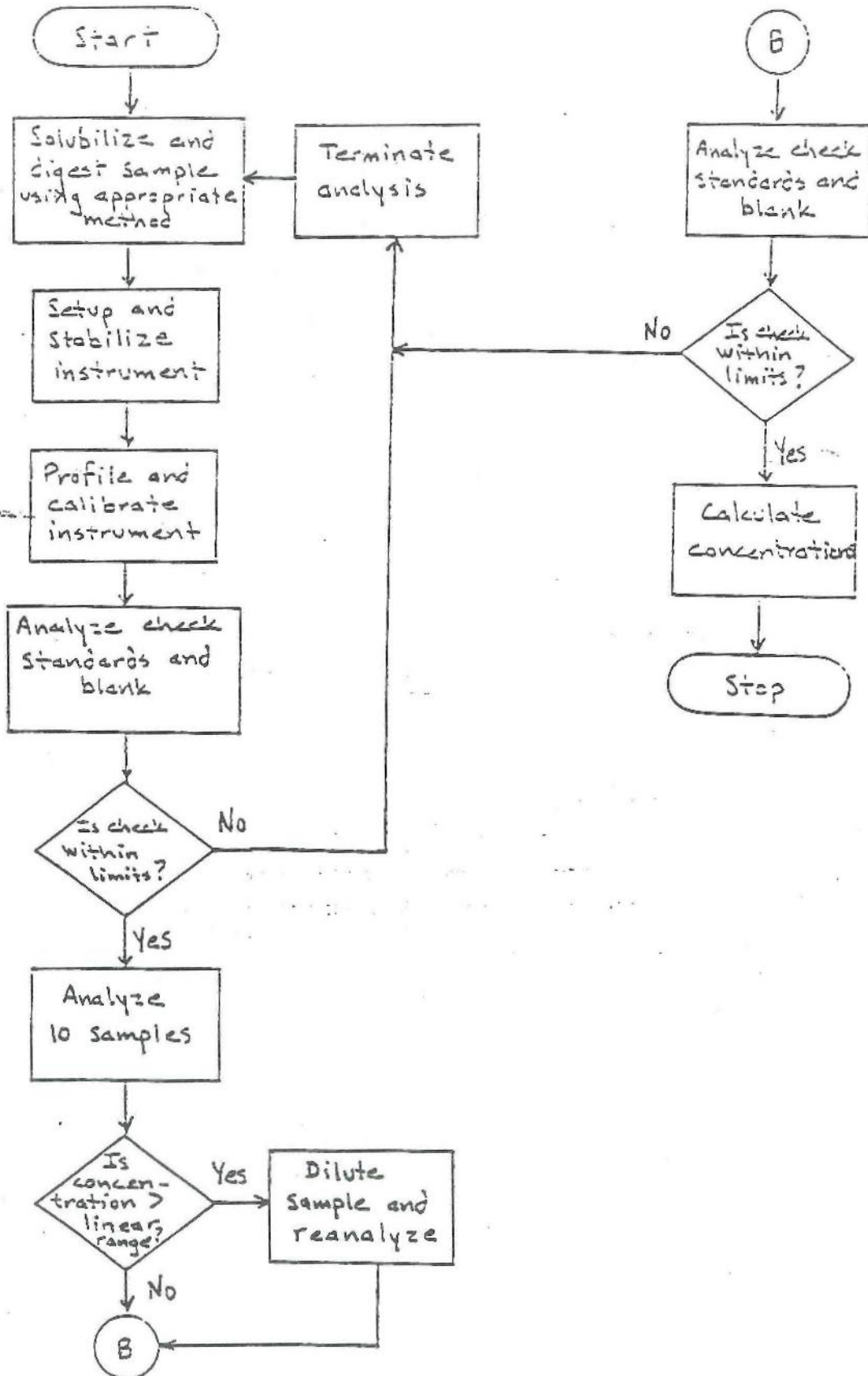
### DATA DELIVERABLES:

Reports to client will include:

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix
- Laboratory ID#
- Client ID#
- Analytical method #
- Concentrations Determined and resulting EQL's
- ICV, CCV Summary form
- ICB, CCB, Prep Blank Summary form
- ICP Interference Check Sample Summary form
- Spike Sample Recovery form
- Duplicate Sample Summary form
- Laboratory Control Sample Summary form
- All Raw Data

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- Preparation Records



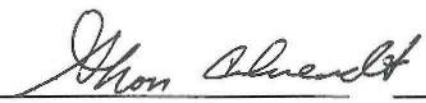
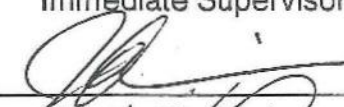
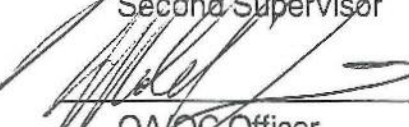

SOP ID: SOP-MET-3005-1  
Rev. Number: 1.2  
Rev. Date: February 6, 1995

## Standard Operating Procedure For Acid Digestion of Aqueous Samples and Extracts For Analysis by Inductively Coupled Argon Plasma Spectroscopy

Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 3005

Revision # 1.2  
Issued: February 6, 1995

	9-26-96
Immediate Supervisor	Date
	9-26-96
Second Supervisor	Date
	9-26-96
QA/QC Officer	Date
	9.26.96
Analyst	Date

Effective: February 6, 1995

### CAUTION

**Disclaimer:** This Standard Operating Procedure has been prepared for the sole use of American Analytical, Inc. and may not be specifically applicable to the activities of other organizations.

Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-3005-1  
Rev. Number: 1.2  
Rev. Date: February 6, 1995

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Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-3005-1  
Rev. Number: 1.2  
Rev. Date: February 6, 1995  
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STANDARD OPERATING PROCEDURE  
ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR ANALYSIS  
BY INDUCTIVELY COUPLED ARGON PLASMA SPECTROSCOPY

REFERENCE:

SW-846, 3rd Edition, method 3005  
USEPA CLP SOW ILM02.0

MATRIX:

Water, Leachate

DETECTION LIMITS:

N/A

RANGE:

N/A

PRINCIPLE, SCOPE AND APPLICATION:

This digestion procedure is used for the preparation of aqueous samples, for analysis by Inductively Coupled Argon Plasma (ICAP) spectroscopy. The following elements can be analyzed using this preparation procedure: Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Vanadium, and Zinc.

The entire sample is acidified to pH <2, with nitric acid, at time of collection. For digestion, the sample is combined with acid and heated until volume is greatly reduced. If needed, the digestate is filtered and brought to volume, and is ready for analysis.

INTERFERENCES AND CORRECTIVE ACTION:

The digestion procedure may not be sufficient to completely break-down some metal complexes. Additional digestion time or a more vigorous digestion may be required to facilitate complete digestion.

Precipitation during digestion may cause a suppression of the total Silver content measured in the analysis. If this occurs, dilute the sample prior to digestion to lower the effective concentration of Silver to < 1 ppm.

Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-3005-1  
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4) Add, under a fume hood, 1ml of concentrated nitric acid and 2.5 ml concentrated hydrochloric acid to the sample.

5) Cover beaker with a watch glass and place on a hot-plate to be heated at 95°C for two hours, or until the sample volume is reduced to 15 to 25 ml. Do not allow the digestate to boil.

6) Cool the digestate to room temperature. If needed, filter the digestate through qualitative filter paper into a graduated cylinder and adjust sample volume to 50 ml with DI water.

#### QUALITY CONTROL:

For each batch of digestions, not to exceed 20, a preparation blank, laboratory control standard, duplicate, and spike will be prepared.

A check blank will be conducted on each new lot of nitric acid and hydrochloric acid prior to use, to verify purity.

#### DATA DELIVERABLES:

All digestion activities will be recorded on a digestion log sheet. A copy of this sheet will be provided with the analytical data package.

SOP ID: SOP-MET-7421-1  
Rev. Number: 2.1  
Rev. Date: September 24, 1996

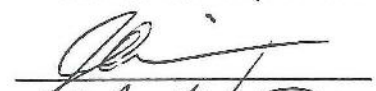
## Standard Operating Procedure For Graphite Furnace Atomic Absorption Analysis of Lead

Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 7421

Revision # 2.1  
Issued: September 24, 1996

  
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Immediate Supervisor      9-26-96  
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Second Supervisor      9-26-96  
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QA/QC Officer      9-26-96  
Date

  
\_\_\_\_\_  
Analyst      9-26-96  
Date

Effective: September 24, 1996

### CAUTION

**Disclaimer:** This Standard Operating Procedure has been prepared for the sole use of American Analytical, Inc. and may not be specifically applicable to the activities of other organizations.

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Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-7421-1  
Rev. Number: 2.1  
Rev. Date: September 24, 1996  
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**STANDARD OPERATING PROCEDURE**  
**GRAPHITE FURNACE ATOMIC ABSORPTION ANALYSIS OF LEAD**

**LOCATION:**

Metals, Metals Laboratory

**REFERENCE:**

SW-846, 3rd Edition, Method 7421

**MATRIX:**

Water, Leachate

**DETECTION LIMITS:**

EQL = 5.0 ug/ L; MDL = 0.499 ug/ L

**RANGE:**

5.0 ug/L to 100 ug/L without dilution  
500 ug/kg to 10000 ug/kg without dilution

**PRINCIPLE, SCOPE AND APPLICATION:**

Lead in solution may be readily determined by graphite furnace atomic absorption spectroscopy. The method is simple, rapid, and applicable to a variety of matrices. Samples for totals analysis require digestion prior to analysis.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. When using furnace techniques the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects.

When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the discharge lamp, and a photosensitive device measures the attenuated transmitted radiation.

### INTERFERENCES AND CORRECTIVE ACTION:

Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

- 1) Successively dilute and reanalyze the samples to eliminate interferences.
- 2) Analyze the sample by method of standard additions while noticing the precautions and limitations of its use.

Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. Background correction may also compensate for nonspecific broad-band absorption interference.

Continuous background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g. Zeeman background correction.

Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilization step. If another acid in addition to  $\text{HNO}_3$  is required, minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Pipet tips are a frequent source of contamination. If contamination is suspected, the tips should be soaked with 1:5 nitric acid and rinsed thoroughly with DI water.

### SAFETY PRECAUTIONS:

Lab coats and safety goggles are to be worn while working with samples, especially during digestion procedures. All instrument vapors are to be vented to the exterior of the building, and all digestion are to occur under a fume hood. CAUTION: Lead is extremely toxic! Handle with care. Refer to MSDS for any inquiries about reagents or chemicals used in this test.

### SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Aqueous and leachate samples are to be collected in 500 mL plastic containers with teflon lined lids, and preserved to  $\text{pH} \leq 2$  with nitric acid. Samples should be labeled as being preserved. Nonaqueous samples are to be collected in 4 oz. squat jars with teflon lined lids and cooled to  $4^\circ\text{C}$  until digestion. Samples must be analysed within 6 months of collection.

### APPARATUS:

- 1) Varian SpectrAA 400 with double beam, grating monochromator, photomultiplier detector, adjustable slits, wavelength range of 190 to 800 nm, Zeeman background correction, and interfaced with an IBM computer and dot matrix printer.
- 2) Zeeman Graphite Tube Atomizer provides power to furnace and spectrophotometer. Allows use of two gasses, and requires cooling water. Provides temperature range of 40 - 3000 C and heating times of 0 - 500 seconds. Provides gas control between 0 and 3.1 L/ min.
- 3) Autosampler with capability of running 45 samples including check standards. Dispenses volumes from 1 to 40 uL.

- 4) IBM PS/2 Model 30 computer, controls operation of spectrophotometer and provides data manipulation and reporting of samples calculations.
- 5) Citizen dot matrix printer, prints calibration and sample results.
- 6) Class A volumetric flasks
- 8) Pipets: Microliter, with disposable tips. Sizes can range from 5 to 100 uL as required. Pipet tips should be checked as a possible source of contamination prior to their use.
- 9) Class A volumetric pipets
- 10) Analytical balance
- 11) Disposable glass serological pipets

#### ROUTINE MAINTENANCE:

Gasses are checked daily to insure adequate pressure. The autosampler parts are checked daily. Furnace optics are cleaned twice weekly. Plumbing connections, and the furnace are checked as needed. Electrodes are changed as needed. Graphite tube is changed as needed.

#### REAGENTS AND CALIBRATION STANDARDS:

- 1) Deionized water - Type II
- 2) Nitric Acid - concentrated, trace metals grade (Fisher, AS09-212)
- 3) Furnace Stock Calibration Standard: Using a Class A volumetric pipet, dilute 2.0 mL lead stock (Spex, PLB2-2y), and 4.0 mL concentrated nitric acid, to 200 mL with DI water in a volumetric flask. This will result in a final concentration of 10 ppm lead. Dilute stock calibration standard 1:99 with DI water for daily calibration.
- 4) Furnace ICV/CCV Solutions: Using a 100 uL micropipet and a 10 mL disposable glass serological pipet, transfer 0.10 mL QC-19 stock (Spex, QC-19, 100 ppm) and 2.0 mL concentrated nitric acid to a 100 mL Class A volumetric flask. Bring to volume. This will result in a 100 ppb final concentration. Dilute 1:1 for a 50 ppb working solution.

5) Lead Modifier: Using a Class A volumetric pipet, transfer 1.00 mL phosphoric acid into a Class A volumetric flask, partially filled with DI water. Bring to volume.

#### CALIBRATION PROCEDURES:

A curve consisting of 5 standards and a blank is analyzed at the beginning of each run. The curve must demonstrate a correlation coefficient of  $\geq 0.995$  to be valid. An ICV followed by an ICB are analyzed prior to sample analysis. The ICV must recover within 20 % of true value, and the ICB must show results less than the EQL. After every 10 samples, and at the conclusion of the run, a CCV and CCB are analyzed. The CCB and CCV must meet the above stated criteria for the ICV and ICB.

#### SAMPLE PREPARATION:

Aqueous: See SOP-MET-3020-1

Nonaqueous: See SOP-MET-3050-1

#### ANALYSIS PROCEDURE:

- 1) Turn on monitor, computer, Spectra AA 400, Zeeman, Graphic Tube Atomizer, T & A cooling unit, hood printer, Argon gas at its source. Press F10 (index) on computer keyboard. Type 10, press F6 (new page) press F1 (clear sequence). Type the number of the program to be run. Press F6, the program is loaded and the correct lamp is automatically moved into position.
- 2) Swing toggle lever clockwise to release the furnace right hand housing. Clean furnace housing using a cotton swab and isopropyl rubbing alcohol. Clean a graphite tube and its platform using a Kimwipe. Position graphite platform inside the plateau tubes so that it is perpendicular to the sample injection hole of the tube. Place the graphite tube in the furnace housing being careful to align the sample introduction hole in the graphite tube to the center of the furnace chimney. Swing the toggle lever counter-clockwise in order to close the righthand housing onto the tube now positioned inside the furnace housing.
- 3) Remove rinse bottle and fill to the line with DI H<sub>2</sub>O. Clean the blank, modifier, and standard cups with DI H<sub>2</sub>O and 1:1 nitric acid. Fill and place these cups in their labeled positions on the autosampler tray.
- 4) Press F10 (index). Type 8 and press F6 (new page). Press F2 (align sampler) twice. The sampling arm will move from its rinse position to the sample 1

position and then to the sample introduction hole in the graphite tube. Adjust the position of the auto sampler capillary tube inside the hole in the graphite tube so that it is in the center of this hole. Use the backwards and forward adjuster along with the sideways adjuster to accomplish the correct positioning.

5) Open syringe compartment door. Put the syringe clear of its mounting and remove the plunger from the syringe. While holding a tissue beneath the syringe press F3 (rinse). Liquid will emerge along with any air bubbles present in the line. Press F3 (rinse) again, and while solution is dripping from the syringe, carefully insert the plunger into the syringe. Reinsert the syringe assembly into its housing and close the compartment door.

6) Press F10 (index). Type 18 and Press F6 (new page). Press F4 (tube clean). The furnace will heat and clean the graphite tube. Press shift and F11 (start GTA). A trial start will begin. Watch the sampler to ensure it pulls up blank and modifier solution into the capillary and is properly injected onto the platform inside the graphite tube. Swing the mirror assembly counter-clockwise to force it in the path of the UV light and thus putting in view the position of the capillary while inside the graphite tube. Ensure that the droplet is placed correctly in the tube. Allow the temperature program to go to completion and note the analytical signal.

7) Press F10 (index). Type 6 and then F6 (new page). Open the Spectra 400 lamp cover and by turning the two knobs on the left-hand side of the appropriate lamp adjust the angle until the lamp peak wavelength has been found (i.e. the optimization line is at its furthest most position from the left-hand baseline). Note: pressing F1 (rescale) allows the wavelength line that may reach a maximum at the right hand edge of the screen to rescale at a point near the middle of the screen. Once the lamp has sufficiently warmed (approximately 20 minutes from the time of the program) the run can be started.

8) Pour samples to be analyzed into sample cups and place them into the autosampler tray. Record position of samples in tray on sample run list log. Pour check standards into sample cups and place them in their proper positions in the autosampler tray. Press F10 (index). Type 15 and press F6 (new page). Press F11 (start) to begin sample run.

Specific settings for lead:

Lead: Program #4, Matrix Modifier - Lead Modifier

Standard 1 = 5.00 ppb, 2 = 10.0 ppb, 3 = 25.0 ppb, 4 = 50.0 ppb, 5 = 99.00 ppb

ICV = 50.0 ppb, CCV = 50.0 ppb

### QUALITY CONTROL:

All quality control data should be maintained and available for easy reference or inspection.

If 10 or more samples per batch are analyzed, the working standard curve must be verified by running an additional standard at or near the mid-range every 10 samples. Checks must be within  $\pm 20\%$  of true value.

At least one preparatory blank, laboratory standard, spike, and duplicate sample should be run every 20 samples, or with each matrix type to verify precision of the method.

Where the sample matrix is so complex the viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition may be used. (See below).

#### *Method of standard additions*

In the simplest version of this method, equal volumes of sample are added to a DI water blank and to a standard. If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, then the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

- 1) The absorbance plot of sample and standards must be linear over the concentration range of concern. For best results, the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be used.
- 2) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.
- 3) The determination must be free of spectral interference and corrected for nonspecific background interference.

The simplest version of this technique is the single addition method, in which two identical aliquots of the sample solution, each of Volume  $V_x$  are taken. To the first (labeled A) is added a small volume  $V_s$  of a standard analyte solution of concentrate  $c_s$ . to the second (labeled B) is added the same volume  $V_s$  of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration  $c_x$  is calculated:

$$c_x = S_b V_s C_s / (S_a - S_b) V_x$$

where,

$S_a$  and  $S_b$  are the analytical signals (corrected for the blank) of solutions a and b respectively.  $V_s$  and  $c_s$  should be chosen so that  $S_a$  is roughly twice  $S_b$  on the average. It is best if  $V_s$  is made much less than  $V_x$  and thus  $c_s$  is much greater than  $c_x$  to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

#### DATA TREATMENT:

For determination of metal concentration by direct aspiration and furnace, read the metal value in ug/L from the calibration curve or directly from the read-out system of the instrument.

If dilution of sample was required:

$$\text{ug/L metal in sample} = (A) [ (C + B)/C ]$$

where,

A = ug/L of metal in diluted aliquot from calibration curve

B = acid blank matrix used for dilution, mL

C = sample aliquot, mL

For solid samples report all concentrations as ug/kg based on wet weight.  
 Hence:

$$\text{ug/metal/kg sample} = \frac{A \times V}{W}$$

where,

A = ug/L of metal in processed sample from calibration curve

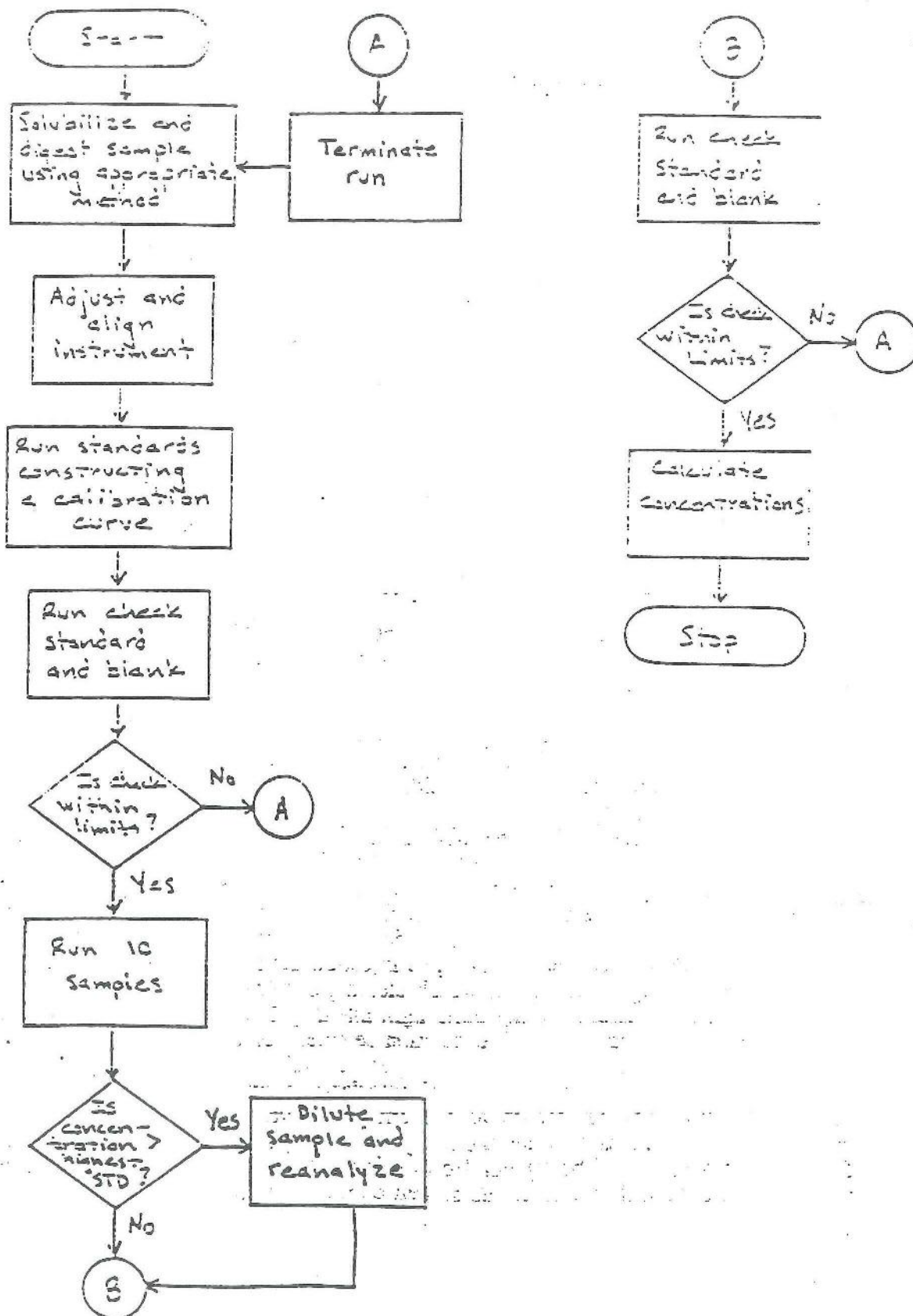
V = final volume of processed sample, ml

W = weight of sample, grams

#### DATA DELIVERABLES:

Reports to clients will include:

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix
- Laboratory ID#
- Client ID#
- Analytical method #
- Concentration determined and resulting EQL
- ICV, CCV, summary form
- ICB, CCB, prep blank summary form
- Spike sample recovery form
- Laboratory control sample summary form
- All raw data
- Preparation records



Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-3020-1  
Rev. Number: 1.1  
Rev. Date: February 6, 1995

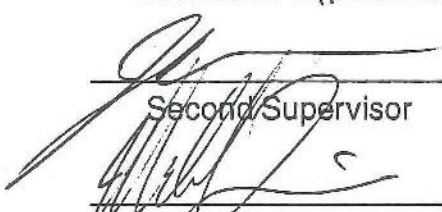
## Standard Operating Procedure For Acid Digestion of Aqueous Samples and Extracts For Analysis by Graphite Furnace Atomic Absorption Spectroscopy

Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Metals, Metals Laboratory

SW-846, 3rd Edition, Method 3020

Revision # 1.1  
Issued: February 6, 1995

 9-26-96  
\_\_\_\_\_  
Immediate Supervisor Date

 9-26-96  
\_\_\_\_\_  
Second Supervisor Date

 9-26-96  
\_\_\_\_\_  
QA/QC Officer Date

 9-26-96  
\_\_\_\_\_  
Analyst Date

Effective: February 6, 1995

### CAUTION

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Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-3020-1  
Rev. Number: 1.1  
Rev. Date: February 6, 1995

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Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-3020-1  
Rev. Number: 1.1  
Rev. Date: February 6, 1995  
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STANDARD OPERATING PROCEDURE  
ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR ANALYSIS  
BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROSCOPY

REFERENCE:

SW-846, 3rd Edition, methods 3020A, 7060, and 7740  
USEPA CLP SOW ILM02.0

MATRIX:

Water, Leachate

DETECTION LIMITS:

N/A

RANGE:

N/A

PRINCIPLE, SCOPE AND APPLICATION:

This digestion procedure is used for the preparation of aqueous samples, for analysis by Graphite Furnace Atomic Absorption spectroscopy. The following elements can be analyzed using this preparation procedure: Arsenic, Beryllium, Cadmium, Chromium, Cobalt, Lead, Molybdenum, Selenium, Thallium, and Vanadium.

This procedure is used to determine the total amount of the metal of concern in the samples submitted. A mixture of nitric acid, hydrogen peroxide and the sample is heated, initiating the digestion process. After the digestate has been brought to a low volume, it is cooled and brought to volume in dilute nitric acid, such that the final digestate contains 2 % (V/V) nitric acid and hydrogen peroxide.

INTERFERENCES AND CORRECTIVE ACTION:

Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilization step. If another acid in addition to nitric acid is required, minimum quantities should be used. This applies particularly to hydrochloric and to a lesser extent sulfuric and phosphoric acids.

Cross-contamination and contamination of the sample can be a major source of error because of the extreme sensitivities achieved with the furnace. The sample preparation work area should be kept scrupulously clean. All glassware should be cleaned as

Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-3020-1  
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5) Cover beaker with a watch glass and place on a hot-plate to be heated at 95°C for two hours, or until the sample volume is reduced to 15 to 25 ml. Do not allow the digestate to boil.

6) Cool the digestate to room temperature. If needed, filter the digestate through qualitative filter paper into a graduated cylinder and adjust sample volume to 50 ml with DI water.

#### QUALITY CONTROL:

For each batch of digestions, not to exceed 20, a preparation blank, laboratory control standard, duplicate, and spike will be prepared.

A check blank will be conducted on each new lot of nitric acid and peroxide prior to use, to verify purity.

#### DATA DELIVERABLES:

All digestion activities will be recorded on a digestion log sheet. A copy of this sheet will be provided with the analytical data package.

SOP ID: SOP-MET-7740-1  
Rev. Number: 3.1  
Rev. Date: February 6, 1995


## Standard Operating Procedure Atomic Absorption Analysis of Selenium

Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Metals, Metals Laboratory

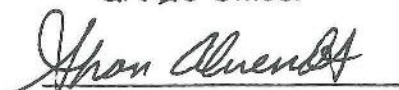
SW-846, 3rd Edition, Method 7740

Revision # 3.1  
Issued: February 6, 1995

  
Immediate Supervisor      9-26-96  
Date

  
Second Supervisor      9-26-96  
Date

  
QA/QC Officer      9-26-96  
Date

  
Analyst      9-26-96  
Date

Effective: February 6, 1995

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Bethlehem Steel Corporation  
SOP-MET-7740-1  
Number: 3.1  
Date: February 6, 1995  
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## STANDARD OPERATING PROCEDURE ATOMIC ABSORPTION ANALYSIS OF SELENIUM

### REFERENCE:

SW-846, 3rd Edition, Method 7060

### MATRIX:

Water, Leachate, Soil, Sludge, Solids

### DETECTION LIMIT:

EQL = 5 ug/L; MDL = 1.6 ug/L

### RANGE:

5 ug/L to 50 ug/L without dilution

500 ug/kg to 5000 ug/kg without dilution

### PRINCIPLE, SCOPE, AND APPLICATION:

Selenium in solution may be readily determined by graphite furnace atomic absorption spectroscopy. The method is simple, rapid, and applicable to a variety of matrices. Samples for totals analysis require digestion prior to analysis.

Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and models of atomic absorption spectrophotometers. When using furnace techniques the analyst should be cautioned as to possible chemical reactions occurring at elevated temperatures which may result in either suppression or enhancement of the analysis element. To ensure valid data with furnace techniques, the analyst must examine each matrix for interference effects.

When using the furnace technique in conjunction with an atomic absorption spectrophotometer, a representative aliquot of a sample is placed in the graphite tube in the furnace, evaporated to dryness, charred, and atomized. Radiation from a given excited element is passed through the vapor containing ground-state atoms of that element. The metal atoms to be measured are placed in the beam of radiation by increasing the temperature of the furnace, thereby causing the injected specimen to be volatilized. A monochromator isolates the discharge lamp, and a photosensitive device measures the attenuated transmitted radiation.

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### INTERFERENCES AND CORRECTIVE ACTION:

Although the problem of oxide formation is greatly reduced with furnace procedures because atomization occurs in an inert atmosphere, the technique is still subject to chemical interferences. The composition of the sample matrix can have a major effect on the analysis. It is those effects which must be determined and taken into consideration in the analysis of each different matrix encountered. To help verify the absence of matrix or chemical interference, the serial dilution technique may be used. Those samples which indicate the presence of interference should be treated in one or more of the following ways:

- (1) Successively dilute and reanalyze the samples to eliminate interferences.
- (2) Analyze the sample by method of standard additions while noticing the precautions and limitations of its use.

Gases generated in the furnace during atomization may have molecular absorption bands encompassing the analytical wavelength. Background correction may also compensate for nonspecific broad-band absorption interference.

Continuous background correction cannot correct for all types of background interference. When the background interference cannot be compensated for, chemically remove the analyte or use an alternate form of background correction, e.g. Zeeman background correction.

Interference from a smoke-producing sample matrix can sometimes be reduced by extending the charring time at a higher temperature or utilizing an ashing cycle in the presence of air. Care must be taken, however, to prevent loss of the analyte.

Samples containing large amounts of organic materials should be oxidized by conventional acid digestion before being placed in the furnace. In this way, broad-band absorption will be minimized.

Anion interference studies in the graphite furnace indicate that, under conditions other than isothermal, the nitrate anion is preferred. Therefore, nitric acid is preferable for any digestion or solubilization step. If another acid in addition to  $\text{HNO}_3$  is required, minimum amount should be used. This applies particularly to hydrochloric and to a lesser extent to sulfuric and phosphoric acids.

Cross-contamination and contamination of the sample can be a major source of error. The sample preparation work area should be kept scrupulously clean. Pipet tips are a frequent source of contamination. If contamination is suspected, the tips should be soaked with 1:5 nitric acid and rinsed thoroughly with DI water.

### SAFETY PRECAUTIONS:

Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-7740-1  
Rev. Number: 3.1  
Rev. Date: February 6, 1995  
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## REAGENTS AND CALIBRATION STANDARDS:

Deionized water - Type II

Nitric Acid - concentrated, trace metals grade

Hydrochloric Acid(1:1) - trace metals grade. Prepare by combining equal portions of concentrated HCl with DI water.

Furnace stock calibration standard: Dilute 1.0 ml Selenium Stock(Fisher - 1000 ppm), and 4.0 ml concentrated nitric acid, to 200 ml with DI water in a volumetric flask. This will result in a final concentration of 5 ppm Selenium. Add Dilute stock calibration standard 1:99 with DI water for daily calibration.

Furnace ICV/CCV Solutions: Dilute 0.10 ml QC-19 Stock(SPEX - 100 ppm) and 2.0 ml concentrated nitric acid to 100 ml with DI water in a volumetric flask. This will result in a 100 ppb final concentration. Dilute 1:3 for a working concentration of 25 ppb.

Nickel Nitrate Modifier: 0.4950 g  $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  plus 5.0 ml concentrated nitric acid. Dilute to 100 ml with DI water in a volumetric flask.

## CALIBRATION PROCEDURES:

A curve consisting of 4 standards and a blank is analyzed at the beginning of each run. The curve must demonstrate a correlation coefficient of  $\geq 0.995$  to be valid. An ICV followed by an ICB are analyzed prior to sample analysis. The ICV must recover within 20 % of true value, and the ICB must show results less than the EQL. After every 10 samples, and at the conclusion of the run, a CCV and CCB are analyzed. The CCV and CCB must meet the above stated criteria for the ICV and ICB.

## SAMPLE PREPARATION:

Aqueous: See SOP-MET-3020-1

Non-aqueous: See SOP-MET-3050-1

## ANALYSIS PROCEDURE:

- 1.) Turn on monitor, computer, Spectra AA-400, Zeeman, Graphic Tube Atomizer, T & A cooling unit, hood printer, Argon gas at its source. Press F10 (index) on computer keyboard. Type 10, press F-6 (new page) press F1 (clear sequence). Type the number of the program to be run. Press F6, the program is loaded and the correct lamp is automatically moved into position.
- 2.) Swing toggle lever clockwise to release the furnace right hand housing. Clean furnace housing using a cotton swap and isopropyl rubbing alcohol. Clean a graphite tube and it's platform using a Kim wipe. Position graphite platform

Client: Bethlehem Steel Corporation  
SOP ID: SOP-MET-7740-1  
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Selenium: Program #10, Matrix Modifier - Nickel Nitrate  
Standard 1 = 5ppb, 2 = 10ppb, 3 = 25ppb, 4 = 50ppb  
ICV = 25ppb, CCV = 25ppb

### QUALITY CONTROL:

All quality control data should be maintained and available for easy reference or inspection.

If 10 or more samples per batch are analyzed, the working standard curve must be verified by running an additional standard at or near the mid-range every 10 samples. Checks must be within  $\pm 20\%$  of true value.

At least one spike and one spike duplicate sample should be run every 20 samples, or with each matrix type to verify precision of the method.

Where the sample matrix is so complex the viscosity, surface tension and components cannot be accurately matched with standards, the method of standard addition may be used. (See below.)

#### *Method of standard additions:*

In the simplest version of this method, equal volumes of sample are added to a DI water blank and to a standard. If a higher degree of accuracy is required, more than one addition should be made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, then point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

The method of standard additions can be very useful; however, for the results to be valid the following limitations must be taken into consideration:

(a) The absorbance plot of sample and standards must be linear over the concentration range of concern. For best results, the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%), caution should be exercised.

(b) The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

(c) The determination must be free of spectral interference and corrected for nonspecific background interference.

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of Volume  $V_x$ , are taken. To the first

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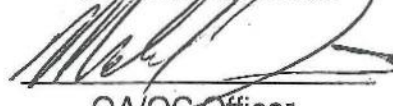

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix
- Laboratory ID#
- Client ID#
- Analytical method #
- Concentration Determined and resulting EQL
- ICV, CCV Summary form
- ICB, CCB, Prep Blank Summary form
- Spike Sample Recovery form
- Laboratory Control Sample Summary form
- All Raw Data
- Preparation Records

## Standard Operating Procedure For Volatile Organics Compounds (VOC)

Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Organics, Organics Laboratory & Main Laboratory

SW-846, 3rd Edition, Method 8260A

Revision # 2.1  
Issued: September 24, 1996

 Immediate Supervisor	<u>11-6-96</u> Date
 Second Supervisor	<u>11/6/96</u> Date
 QA/QC Officer	<u>11-06-96</u> Date
 Analyst	<u>11/6/96</u> Date

Effective: September 24, 1996

### CAUTION

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SOP ID: SOP-ORG-8260A-1  
Rev. Number: 2.1  
Rev. Date: September 24, 1996  
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**STANDARD OPERATING PROCEDURE**  
**VOLATILE ORGANIC COMPOUNDS (VOC)**

**LOCATION:**

Organics, Organics Laboratory

**REFERENCE:**

SW-846, 3rd Edition, Method 8260A  
U.S. EPA Method 624

**MATRIX:**

Water, Leachate, Soil, Solids, Sludge

**DETECTION LIMITS:**

For EQL's and MDL's see Table 1-1 attached.

**RANGE:**

Aqueous: 5 ug/L to 200 ug/L without dilution

Non-aqueous: 5 ug/kg to 200 ug/kg without dilution

**PRINCIPLE, SCOPE AND APPLICATION:**

Volatile organic compounds are extracted from a water matrix using the purge and trap method and analyzed on a computer controlled GC/MS system following the criteria established in SW-846 Method 8260.

**INTERFERENCES AND CORRECTIVE ACTION:**

Since SW-846 Method 8260 uses extracted ion counts and internal standards, interferences are kept to a minimum.

**SAFETY PRECAUTIONS:**

Lab coats and safety glasses are to be worn while working with samples. Standards are stored in crimp-top vials in a freezer.

**SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:**

SOP ID: SOP-ORG-8260A-1  
Rev. Number: 2.1  
Rev. Date: September 24, 1996  
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Aqueous samples should be collected in 40 ml vials with teflon lined lids. Care should be taken to eliminate all head-space from the vials. Non-aqueous samples should be collected in a 4 oz. squat jar. Aqueous and non-aqueous samples should be cooled to 4 °C until analysis. Holding times are 7 days for unpreserved waters and 14 days for soils and preserved waters.

#### APPARATUS:

Tekmar ALS 2016 equipped with 25 mL sparger tubes.

Tekmar LSC 2000 with MCM and cryofocusing module configured as follows:

Standby - 42°  
Purge - 12 minutes  
Dry Purge - 1 minute  
MCM cool down - 36 °C  
Cryofocusing cool down - -155 °C  
Desorb - 4 minutes at 250 °C  
Inject - 1.5 minutes at 160 °C  
Bake - 4 minutes at 260 °C

HP 5890 Series II Gas Chromatograph configured as follows:

Initial temp. - 10 °C  
Initial time - 1 minute  
Rate - 6 °C/minute  
Final temp. - 124 °C  
Final time - 0 minutes

HP 5971A Mass selective Detector configured as follows:

Scan Mode  
Mass range 35 to 350  
Sampling 2  
Threshold - 500

HP Vectra 386/25 computer with MS Chemstation software, monitor and LaserJet II

10 uL, 500 uL and 5.0 mL syringe

2 mL vials with crimp tops

### ROUTINE MAINTENANCE:

The VOC purge glassware is cleaned after each run. A daily check of the carrier gas is done and the tank is changed when the pressure reaches 300 psi. Pump leaks are checked for quarterly. Changing septa, changing gas filters, changing trap on Tekmar, changing GC column, cleaning MS source, checking septa for leaks, checking gas flow, cutting capillary column, and replacing liner are all done as needed.

### REAGENTS AND CALIBRATION STANDARDS:

- a) M-8240A 10X, Accustandard(or equivalent)  
2000 ug/ml each non-gas component in methanol
- b) M-601-B 10X Gasses, Accustandard(or equivalent)  
2000 ug/ml each gas component in methanol
- c) M-502-28 10X, Accustandard(or equivalent)  
2000 ug/ml cis-1,2-dichloroethene in methanol
- d) CLP-PI 2.5X, Purgeable internal standard, Accustandard(or equivalent)  
2500 ug/ml each component in methanol
- e) M-8240/60-SS-10X, Purgeable surrogate standard, Accustandard (or equivalent)  
2500 ug/ml each component in methanol
- f) CLP-003R 10X, Purgeable organic matrix spiking solution, Accustandard  
(or equivalent) 2500 ug/ml each component in methanol
- g) Methanol, purge & trap grade
- h) Organic free water
- i) VOC working calibration standard
  - 1) 50 ug/ml each component
  - 2) preparation procedure: Place 925 ul MeOH in a 2 ml crimp-top vial and add 25 ul of each target compound mix(a, b, and c). Cap vial and store in freezer.
- j) VOC internal standard working solution
  - 1) 50 ug/ml each component
  - 2) preparation procedure: Place 980 ul MeOH in a 2 ml crimp-top vial and add 20 ul of purgeable internal standard mix. Cap vial and store in freezer.
- k) VOC surrogate working solution

1) 50 ug/ml each component

2) preparation procedure: Place 980 ul MeOH in a 2 ml crimp-top vial and add 20 ul of purgeable surrogate standard mix. Cap vial and store in freezer.

1) VOC matrix spike working solution

1) 50 ug/ml each component

2) preparation procedure: Place 980 ul MeOH in a 2 ml crimp-top vial and add 20 ul of purgeable organic matrix spiking mix. Cap vial and store in freezer.

### CALIBRATION PROCEDURES:

An initial 5 pt. calibration is prepared using 1.0 uL, 2.0 uL, 5.0 uL, 10 uL and 20 uL of the working calibration standard added to 5.0 mL of organic-free (DI) water with 5 uL of the working internal standard and surrogate standard. This will correspond to 10 ug/ml, 20 ug/ml, 50 ug/ml, 100 ug/ml and 200 ug/ml of each component except the internal standards and surrogates which will remain at 50 ug/ml. Response factors (RF) for each component are then calculated using the internal standards (see Calculations section).

Prior to analyzing samples, 50 ng of BFB must be analyzed and the resulting spectra must meet the following criteria:

#### Mass Ion Abundance Criteria

50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

This is done by purging 1.0 uL of the Volatiles Surrogate Working Mix in 5 mL of DI water and analyzing on the GC/MS system using the BFB method built into the software. The above criteria must be demonstrated for each 12-hour shift.

In addition to a BFB check every 12 hours, a continuing calibration check must also be run to show the system is operating correctly and the initial calibration is still valid. This is done by purging 5 uL of the VOC working calibration standard, 5 ul of the VOC internal standard working mix, and 5 ul of the VOC surrogate working mix, in 5 ml of DI water and analyzing on the GC/MS system using the 624 Method built into the software. For specific SPCC and CCC compounds and criteria, see Table 6-1 attached.

After the SPCC and CCC have been verified the analyst must analyze a blank sample of 5.0 mL DI water 5 ul of the internal standard and surrogate to show there is not contamination.

### SAMPLE PREPARATION:

#### Aqueous:

- (1) Withdraw 5.5 mL of sample from the sample container using a 5 mL syringe.
- (2) The volume is then adjusted to 5.0 ml and 5 ul of the internal standard and surrogate mixes are added with the 10 uL syringe.
- (3) The contents of the syringe are then transferred to a sparger tube on the ALS 2016 using the L'uer-Lok and valves on the front of the ALS 2016.
- (4) Volatile compounds are extracted by purging with helium.
- (5) The volatile compounds are collected on a trap in the LSC 2000 until they are ready to be desorbed into cryofocusing mount atop the GC.

### ANALYSIS PROCEDURE:

From the cryofocusing mount the volatile compounds are injected directly onto the column. At this time the computer begins to generate a chromatogram of the total amount of ions that pass through the detector. At the end of the run the computer will extract the necessary target ions from the total chromatogram and using the internal standards and RF values from the initial calibration it will generate a report quantifying the sample.

#### Table 1:

- (1) Click on *tune MS* in main menu.
- (2) Click on *Target Tune* under Tune MS
- (3) Click on *tune* in target tune menu
- (4) Click on *BFB tune*
- (5) After tune is complete, double click on left box in Target Tune menu to exit. Save tune file when prompted to.
- (6) Click on *Methods* in main menu
- (7) Click on *Load and Run Method* under methods.
- (8) Click on *BFB624.m* to load method for BFB tune analysis
- (9) Enter data file name as BFB and date.d (e.g. on June 6 enter BFB0706.d).
- (10) Enter operator's name
- (11) Enter sample name as BFB tune for (date)
- (12) Enter miscellaneous information of 50 ng BFB.

- (13) Place 5 mL DI water and 1.0 uL of working surrogate mix in a sparger tube on position 1 of the ALS 2016.
- (14) Press start on the LSC 2000
- (15) Allow method to run and generate a report.
- (16) The report will indicate whether or not the BFB spectrum has passed. If it does not pass, the operator must enter manual tuning, adjust various mass spectrometer parameters (lenses) within the tune file, and perform the tuning process again. The tune should pass before any samples are analyzed.

Table 2:

- (1) Run method 624 by clicking on *load and run method* under method in main menu and then clicking on *624.d*.
- (2) Enter *SPCC and (date).d* for data file name
- (3) Enter operator's name
- (4) Enter sample name as *Daily Calibration Check for (date)*
- (5) Place 5 mL DI into syringe and add 5 uL each of the VOC working standard, internal standard and surrogate. (final conc: 50 ug/mL each).
- (6) Transfer sample into sparger vessel using valves and ALS 2016.
- (7) Press *start* on LSC 2000.
- (8) Click on *run method* to begin analysis.
- (9) Allow to run, retrieve report, check to see that all expected compounds have been calculated at approximately correct concentrations.
- (10) Calculate RF values and % drift values for all system performance check compounds and calibration check compounds according to method 8260 in the SW-846 manual.

Table 3:

- (1) Withdraw 5 ml of sample with a gas-tight syringe and add 5.0 ul of surrogate and internal standard.
- (2) Place sample with surrogates and standards into a clean sparger tube by using the valves on the ALS 2016.
- (3) Press *start* on LSC 2000.

- (4) Click on *run method* in chemstation to enter sample information.
- (5) Enter *s*(last 3 digits of sample log number)and *date.d* for the file name.
- (6) Enter sample amount under sample name.
- (7) Click on *run method* to start analysis and allow to generate a report.

### QUALITY CONTROL:

A matrix spike and a matrix spike duplicate are required to be purged and analyzed for every batch ( batch not to exceed 20 samples). In addition a surrogate is spiked into the blank, matrix spike, matrix spike duplicate, and all the samples. The limits for the spiking compounds and surrogate recoveries are listed on Table 3-1 attached.

### DATA TREATMENT:

The computer report will show the ng/ml of VOC's found in the sample.

$$RF = (A_X C_{is}) / (A_{is} C_X)$$

where,

$A_X$  = Area of the characteristic ion of the compound being measured

$A_{is}$  = Area of the characteristic ion for the specific internal standard

$C_{is}$  = Concentration of the specific internal standard

$C_X$  = Concentration of the compound being measured

$$\% \text{ drift} = \frac{C_I - C_c}{C_I} \times 100$$

where,

$C_I$  = Calibration check compound standard concentration

$C_c$  = Measured concentration

$$\text{concentration (ug/L)} = \frac{(A_X)(I_s)}{(A_{is})(RF)(V_o)} \times DF$$

where,

$A_X$  = Area of the characteristic ion for compound being measured

$I_s$  = Amount of internal standard injected (ng)

$A_{is}$  = Area of characteristic ion for the internal standard

RF = Initial average response factor for compound being measured

$V_0$  = Volume of water purged (mL)

DF = Dilution factor

\*  $\frac{(A_x)(I_s)}{(A_{is})(RF)}$  is calculated by computer

#### DATA DELIVERABLES:

Reports to client will include:

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix
- Laboratory I.D. #
- Client I.D. #
- Analytical method and #
- Blank data
- Concentrations determined and resulting limits
- Surrogate summary form
- Matrix spike summary form
- Blank summary form
- Tune check summary form
- Initial calibration summary form
- Continuing calibration summary form
- IS area/retention time summary form
- All raw data
- Target spectra for all positive hits with corresponding reference spectra
- Preparation records


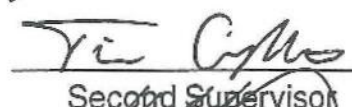


Client: IDEM-BAA 95-30  
SOP ID: SOP-ORG-8270B-1  
Rev. Number: 3.0  
Rev. Date: November 6, 1996  
Page: 1

## Standard Operating Procedure For Semi-Volatile Organic Compounds (SVOC)

Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Organics, Organics Laboratory & Main Laboratory

SW-846, 3rd Edition, Method 8270B

Revision # 3.0  
Issued: November 6, 1996

 MZ Immediate Supervisor	11-6-96 Date
 Second Supervisor	11/6/96 Date
 QA/QC Officer	11-6-96 Date
 Analyst	11/6/96 Date

Effective: November 6, 1996

### CAUTION

**Disclaimer:** This Standard Operating Procedure has been prepared for the sole use of American Analytical, Inc. and may not be specifically applicable to the activities of other organizations.

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Client: IDEM-BAA 95-30  
SOP ID: SOP-ORG-8270B-1  
Rev. Number: 3.0  
Rev. Date: November 6, 1996  
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**STANDARD OPERATING PROCEDURES**  
**SEMI-VOLATILE ORGANIC COMPOUNDS (SVOC)**

**LOCATION:**

Organics, Organics Laboratory

**REFERENCE:**

SW-846, 3rd Edition, Method 8270B  
U.S. EPA Method 625

**MATRIX:**

Water, Leachate, Soil, Solids, Sludge

**DETECTION LIMITS:**

For EQL's, and MDL's see Table 1-1, attached

**RANGE:**

Aqueous: 10 ug/L to 160 ug/L without dilution  
Non-aqueous: 330 ug/kg to 5300 ug/kg without dilution

**PRINCIPLE, SCOPE AND APPLICATION:**

Semi-volatile organic compounds are extracted from a water matrix using methylene chloride and separatory funnels. After concentration, the extract is injected into the GC/MS system and analysis computer controlled following the criteria established in SW-846 method 8270B.

**INTERFERENCES AND CORRECTIVE ACTION:**

Since SW-846 Method 8270B uses extracted ion counts and internal standards, interferences are kept to a minimum. On occasion, an emulsion may form during

the extraction procedure. These emulsions may require extra standing time, stirring or centrifugation to break them up.

#### SAFETY PRECAUTIONS:

Lab coats and safety glasses are to be worn while working with samples, especially during the extraction procedure. While extracting, separatory funnels must be vented frequently to relieve pressure. Standards are stored in crimp top vials in a freezer and other chemicals are stored in a metal cabinet marked *Flammable*.

#### SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Aqueous samples should be collected in 1 liter amber glass containers with teflon lined lids, and cooled to 4 °C until extraction occurs. Holding times are 7 days for extraction and 40 days for analysis for non-aqueous samples.

#### APPARATUS:

1) HP5890 Series II Gas Chromatograph configured as follows:

Initial Temperature:	40 °C
Initial Time:	4 minutes
Rate:	10 °/ minute
Final Temperature:	270 °C
Final Time	7 minutes

2) HP 5971A Mass Selective Detector configured as follows:

Scan Mode
Mass range 35 to 550
Sampling 2
Threshold 500

3) HP Vectra 386/25 computer with MS Chemstation software, monitor and Laser Jet II printer.

2 L Separatory funnel

Turbo Vap II concentrator tube

1 L and 10 mL graduated cylinders

60 mL funnel

10 uL and 1000 uL syringe

2 mL vials with screw tops

Turbo Vap II evaporator (water bath temperature = 37 °C)  
15 mL test tubes with caps

2 mL vials with crimp tops and inserts

#### ROUTINE MAINTENANCE:

A daily check of the carrier gas is done and the tank is changed when the pressure reaches 300 psi. Pump leaks are checked for quarterly. Changing septa, changing gas filters, changing GC column, cleaning MS source, checking septa for leaks, checking gas flow, cutting capillary column, and replacing liner are all done and recorded in the maintenance log for that system as needed.

#### REAGENTS AND CALIBRATION STANDARDS:

1) HC Base-Neutral Mix 1-Supelco (or equivalent)

bis (2-chloroethoxy) methane	diethylphthalate
bis (2-chloroethyl) ether	dimethylphthalate
bis (2-ethylhexyl) phthalate	di-n-butyl phthalate
bis (2-chloroisopropyl) ether	di-n-octylphthalate
4-bromophenylphenylether	N-nitrosodimethylamine

butylbenzylphthalate                      N-nitrosodi-n-propylamine

4-chlorophenylphenylether              N-nitrosodiphenylamine

2,000 ug/mL each component in methylene chloride

2) HC Base-Neutrals Mix 2-Supelco (or equivalent)

azobenzene hexachlorobenzene

2-chloronaphthalene                      hexachlorobutadiene

1,2-dichlorobenzene                      hexachlorocyclopentadiene

1,3-dichlorobenzene                      hexachloroethane

1,4-dichlorobenzene                      isophorone

2,4-dinitrotoluene                      nitrobenzene

2,6-dinitrotoluene                      1,2,4-trichlorobenzene

2,000 ug/mL each component in methylene chloride

3) HC Phenols Mix-Supelco (or equivalent)

4-chloro-3-methylphenol                      2-nitrophenol

2-chlorophenol                              4-nitrophenol

2,4-dichlorophenol                      pentachlorophenol

2,4-dimethylphenol                      phenol

2,4-dinitrophenol                      2,4,6-trichlorophenol

2-methyl-4,6-dinitrophenol

2,000 ug/ML each component in methylene chloride

4) HC PNA Hydrocarbon Mix-Supelco (or equivalent)

acenaphthene                              chrysene

acenaphthylene	dibenzo (a,h) anthracene
anthracene	fluoranthene
benzo(a)anthracene	fluorene
benzo(b)fluorathene	indeno (1,2,3-c,d) pyrene
benzo (g,h,i) perylene	naphthalene
benzo (a) pyrene	phenanthrene
benzo(k)fluoranthene	pyrene

2,000 ug/mL each component in methylene chloride:benzene (50:50)

5) HC Internal Standard Mix-Supelco (or equivalent)

acenphthene-d <sub>10</sub>	naphthalene-d <sub>8</sub>
chrysene-d <sub>12</sub>	perylene-d <sub>12</sub>
1,4-dichlorobenzene-d <sub>4</sub>	phenanthrene-d <sub>10</sub>

4,000 ug/mL each component in methylene chloride

6) Base-neutral Surrogate Standard Mix - Supelco (or equivalent)

nitrobenzene-d <sub>5</sub>	2-fluorobiphenyl
Terphenyl-d <sub>14</sub>	

1,000 ug/ml each component in methylene chloride

7) Acid Surrogate Standard Mix - Supelco (or equivalent)

phenol-d <sub>6</sub>	2,4,6-tribromophenol
2-fluorophenol	

2,000 ug/mL each component in methylene chloride

8) Acid Spiking Solution - Supelco (or equivalent)

pentachlorophenol	4-chloro-3-methylphenol
phenol	4-nitrophenol
2-chlorophenol	

2,000 ug/mL each component in methylene chloride

9) Base-Neutral Spiking Solution - Supelco (or equivalent)

acenaphthene	1,2,4-trichlorobenzene
N-nitrosodi-n-propylamine	1,4-dichlorobenzene
pyrene	2,4-dinitrotoluene

1,000 ug/mL each component in methylene chloride

10) HC Hazardous Substance Mix-1 - Supelco (or equivalent)

2-methylphenol	2,4,5-trichlorophenol
4-methylphenol	

2,000 ug/mL each component in methylene chloride

11) HC Hazardous Substance Mix 2 - Supelco (or equivalent)

aniline	2-methylnaphthalene
benzyl alcohol	2-nitroaniline
4-chloroaniline	3-nitroaniline
dibenzofuran	4-nitroaniline

2,000 ug/mL each component in methylene chloride

12) Decafluorotriphenylphosphine (DFTPP) - Supelco (or equivalent)

25 mg/mL in methylene chloride

13) Acetone, Methylene Chloride

14) sodium sulfate

15) Decafluorotriphenylphosphine (DFTPP) working

- concentration: 50 ug/mL

- Preparation procedure: place 1.0 mL of methylene chloride into a 2 mL vial and cap. Using a 10 uL syringe, add 2 uL of standard 12 to vial.

16) Level 200 Stock

- Concentration: 200 ug/mL for target compounds and acid surrogates, 100 ug/mL for base/neutral surrogates.

- Preparation procedure: add 500 ul of each target mix and surrogate mix to a 15 ml vial and dilute to a final volume of 5 ml.

17) Level 20, 50, 80, 120, and 160 standards

Conc.: 20- 20 ug/ml targets and acid surrogates.

10 ug/ml base/neutral surrogates.

Conc.: 50- 50 ug/ml targets and acid surrogates.

25 ug/ml base/neutral surrogates.

Conc.: 80- 80 ug/ml targets and acid surrogates.

40 ug/ml base/neutral surrogates.

Conc.: 120-120ug/ml targets and acid surrogates.

60 ug/ml base/neutral surrogates.

Conc.: 160-160ug/ml targets and acid surrogates.

80 ug/ml base/neutral surrogates.

- Preparation procedure

Level 20 is prepared by combining 100 ul of level 200 stock, 890 ul of methylene chloride, and 10 ul of internal standard in a 2 ml crimp-top vial.

Level 50 is prepared by combining 250 ul of level 200 stock, 740 ul of methylene chloride, and 10 ul of internal standard in a 2 ml crimp-top vial.

Level 80 is prepared by combining 400 ul of level 200 stock, 590 ul of methylene chloride, and 10 ul of internal standard in a 2 ml

crimp-top vial.

Level 120 is prepared by combining 600 ul of level 200 stock, 390 ul of methylene chloride, and 10 ul of internal standard in a 2 ml crimp-top vial.

Level 160 is prepared by combining 800 ul of level 200 stock, 190 ul of methylene chloride, and 10 ul of internal standard in a 2 ml crimp-top vial.

18) Surrogate Working Solution

- Concentration: 100 ug/mL acid surrogates  
50 ug/mL base-neutral surrogates
- Preparation procedure: Place 80 ml methylene chloride in a 100 ml volumetric flask and add 5.0 ml of each surrogate standard. Dilute to volume.

19) 1:1 H<sub>2</sub>SO<sub>4</sub> - in a 4 oz. squat jar, add an equal volume of sulfuric acid to a known amount of water.

20) 6N NaOH - Dissolve 24 g of sodium hydroxide pellets into 100 mLs of DI water

CALIBRATION PROCEDURES:

An initial 5 pt. calibration is prepared using the level 20, 50, 80, 120 and 160 initial calibration standards. 1 uL from each vial is injected. After the five runs have been completed, response factors (RF) can be calculated for each component (see Data Treatment section).

Prior to analysis of initial or continuing calibrations or samples, 1 uL of the working DFTPP solution must be analyzed to show the instrument is tuned to the following criteria:

<u>Mass</u>	<u>Ion Abundance Criteria</u>
51	30-60% of mass 198
98	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198

197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

The above criteria must be demonstrated for each 12 hour shift.

In addition to a DFTPP check every 12 hours, a continuing calibration check must also be run to show the system is operating correctly and the initial calibration is still valid. This is done by injecting 1 uL of the SPCC, CCC working solution into the GC/MS while running the 625 method built into the software. For specific SPCC and CCC compounds and criteria, see Table 6-1 attached.

#### SAMPLE PREPARATION:

See SOP-ORG-3510-1 for aqueous matrices.

#### ANALYSIS PROCEDURE:

Place 200 uL of extract into a vial insert and cap. After adding 2.0 uL of internal standard, the sample is ready to be injected. 1 uL is injected through the septum using the computer controlled auto-sampler. At the end of the run, the computer will extract the necessary target ions - from the total ion chromatogram and using the internal standards and RF values from the initial calibration it will generate a report quantifying the sample.

#### Table 1

- (1) Click on *Tune MS* in main menu.
- (2) Click on *Target Tune* under Tune MS.
- (3) Click on *DFTPP tune*, under tune file.
- (4) After tune is complete, double click on left box in Target Tune menu to exit. Save tune file when prompted to.
- (5) Click on *Methods* in main menu.

- (6) Click on *Load and Run Method* under Methods.
- (7) Click on *DFTPP.m* to load method for DFTPP tune analysis.
- (8) Enter date file name as DFTP and date.d (e.i. on July 6 enter DFTP0706.d).
- (9) Enter operators name.
- (10) Enter sample name as *DFTPP tune for (date)*.
- (11) Click on *Run Method*.
- (12) Allow method to run.
- (13) Check ion abundance at peak apex in data analysis window.

#### Table 2

- (1) Run method 8270.
- (2) Enter *SPCC + date.d* for file name.
- (3) Enter operator's name.
- (4) Enter sample name as *Daily Calibration Check for (date)*.
- (5) Enter miscellaneous information a 1 ul/ from 50 ug/ml continuing calibration surrogate and internal standards.
- (6) Click on run method.
- (7) Allow to run, retrieve report, check to see that all expected compounds have been calculated at approximately correct concentrations.
- (8) Calculate RF values and % drift values for all system performance check compounds and calibration check compounds according to 7.43 and 7.44 of method 8270B in the SW-846 manual.

#### Table 3

- (1) Run method 8270.
- (2) Enter *S(last 3 digits of sample log #) and date* for file name.

- (3) Enter sample information under sample name.
- (4) Click on run method.
- (5) Allow to run, retrieve report.
- (6) Check each detected component to make sure chromatography is clear, that the peaks for each ion of the compound show the same retention time, and that they are in correct proportions. Also check to see that surrogate standards are recovered sufficiently (see Table 3.3 attached).

#### QUALITY CONTROL:

A blank, matrix spike and matrix spike duplicate are required to be extracted and analyzed for every batch (batch not to exceed 20 samples). In addition a surrogate is spiked into the blank, matrix spike, matrix spike duplicate, and all the samples.

#### DATA TREATMENT:

The computer report will show the ng of all compounds found in the sample. This number must be calculated to reflect the volume extracted, final volume of extract (including dilutions) and the amount of sample injected.

$$RF = (A_X C_{IS}) / (A_{IS} C_X)$$

where,

$A_X$  = Area of the characteristic ion for the compound being measured.

$A_{IS}$  = Area of the characteristic ion for the specific internal standard.

$C_{IS}$  = Concentration of the specific internal standard.

$C_X$  = Concentration of the compound being measured.

$$\% \text{ drift} = \frac{C_I - C_C}{C_I} \times 100$$

where,

$C_I$  = Calibration check compound standard concentration

$C_C$  = Measured concentration

$$\text{concentration} = \frac{(A_X)(I_S)(V_t)}{(A_{IS})(RF)(V_O)(V_i)}$$

where,

$A_X$  = Area of characteristic ion for compound being measured.

$I_S$  = Amount of internal standard injected (ng).

$V_t$  = Volume of total extract, taking into account dilution (i.e. a 1-to-10 dilution of a 1 ml extract will mean  $V_t = 10,000$  ul. If half the base-neutral extract and half the acid extract are combined,  $V_t = 2,000$ .

$A_{IS}$  = Area of characteristic ion for the internal standard.

$RF$  = Initial average response factor for compound being measured

$V_O$  = Volume of water extracted (L).

$V_i$  = Volume of extract injected (ul).

#### DATA DELIVERABLES:

Reports to client will include:

- Date of receipt

Client: IDEM-BAA 95-30  
SOP ID: SOP-ORG-8270B-1  
Rev. Number: 3.0  
Rev. Date: November 6, 1996  
Page: 13

- Date of preparation
- Date of analysis
- Analyst
- Matrix
- Laboratory I.D. #
- Client I.D. #
- Analytical method and method #
- Blank data
- Concentrations determined and resulting limits
- Surrogate summary form
- Matrix spike summary form
- Blank summary form
- Tune check summary form
- Initial calibration summary form
- Continuing calibration summary form
- IS area/retention time summary form
- All raw data
- Target spectra for all positive hits with corresponding reference spectra
- Preparation records

System: **Department of Labor Corporation**  
SOP ID: **SOP-ORG-3510-1**  
Rev. Number: **1.0**  
Rev. Date: **February 6, 1995**

## **Standard Operating Procedure For Separatory Funnel Liquid-Liquid Extraction**


Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Organics, Main Laboratory


SW-846, 3rd Edition, Method 3510

Revision # 1.0  
Issued: February 6, 1995

  
\_\_\_\_\_  
Immediate Supervisor      9-26-96  
Date

  
\_\_\_\_\_  
Second Supervisor      9-26-96  
Date

  
\_\_\_\_\_  
QA/QC Officer      9-26-96  
Date

  
\_\_\_\_\_  
Analyst      9-26-96  
Date

Effective: February 6, 1995

### **CAUTION**

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Client: Bethlehem Steel Corporation  
SOP ID: SOP-ORG-3510-1  
Rev. Number: 1.0  
Rev. Date: February 6, 1995

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Client: Bethlehem Steel Corporation  
SOP ID: SOP-ORG-3510-1  
Rev. number: 1.0  
Rev. Date: February 6, 1995  
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## STANDARD OPERATING PROCEDURE SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION

### REFERENCE:

SW-846, 3rd Edition, Methods 3510 and 3500

### MATRIX:

Aqueous, Leachate

### DETECTION LIMIT:

N/A

### RANGE:

N/A

### PRINCIPLE, SCOPE, AND APPLICATION:

This procedure is for isolating organic compounds from aqueous samples. This process involves isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures. A measured volume of sample, usually 1 L, at a specific pH ( 5-9 for PCB's, >11 for base/neutral fraction of SVOC and < 2 for acid fraction of SVOC ), is serially extracted with methylene chloride using a separatory funnel. The extract is dried, concentrated, and, as necessary, exchanged into a solvent compatible with analytical method used.

### INTERFERENCES AND CORRECTIVE ACTION:

Samples requiring analysis for volatile organic compounds, can be contaminated by diffusion of volatile organics (particularly chlorofluoro-carbons and methylene chloride) through the sample container septum during shipment and storage. A field blank prepared from organic-free reagent water and carried through sampling and subsequent storage and handling can serve as check on such contamination. Solvents, reagents, glassware, and other processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all glass systems may be required. Interferences coextracted from samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further clean up may be required. Phthalate esters contaminate many

### CALIBRATION PROCEDURES:

N/A

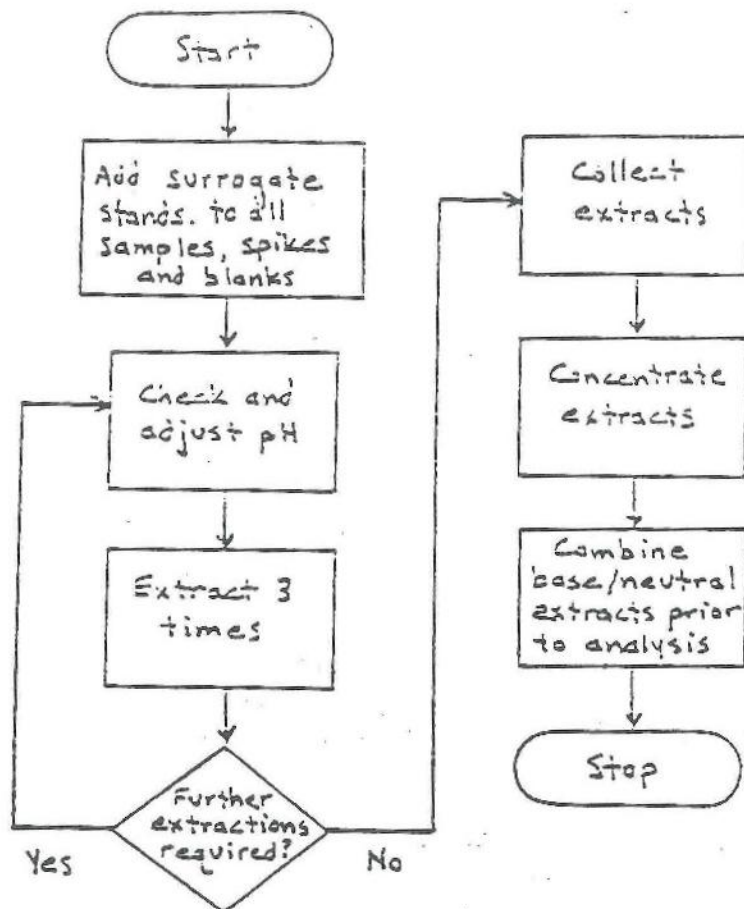
### SAMPLE PREPARATION:

- (1) Rinse all glassware with acetone.
- (2) Triple rinse all glassware and with methylene chloride.
- (3) Transfer sample into separatory funnel.
- (4) Adjust pH if necessary using sulfuric acid or sodium hydroxide
- (5) Add surrogate ( and spikes if needed ) to sample and record type, lot number, and amount added.
- (6) Add methylene chloride to separatory funnel ( between 60 and 65 mL )
- (7) Shake separatory funnel for 2 minutes, venting frequently.
- (8) Prepare funnel filter with glass wool and sodium sulfate.
- (9) Rinse filter with about 20 mL of methylene chloride and discard rinse methylene chloride.
- (10) Drain extract from separatory funnel through filter into concentrator tube.
- (11) Repeat steps (6), (7), and (10) two more times
- (12) Rinse filter to remove any organics still in filter
- (13) Place concentrator tube into Turbo Vap II
- (14) Solvent exchange extract when its volume is below 1 mL and continue to evaporate extract to below 1 mL and remove from Turbo Vap II. If extract does not need a solvent exchange, simply remove extract from Turbo Vap II when extract reaches 1 mL. ( Solvent exchange is adding a different solvent other than the original extraction solvent and evaporating off the original solvent). **CAUTION: DO NOT LET EXTRACT GO DRY!**
- (15) Using the 1 mL syringe, measure and remove extract from concentrator tube and place in appropriate container ( i.e. vials, test tubes, etc. ) labeled with the sample I.D., fraction, volume, parameter, and extraction personnel initials.
- (16) Rinse concentrator tube walls with 2-3 mL of appropriate solvent.
- (17) Using rinse solvent in concentrator tube, adjust volume of extract appropriate volume.
- (18) Cap container of extract.
- (19) Readjust pH of sample for any secondary fraction to be extracted using sulfuric acid or sodium hydroxide.
- (20) Repeat steps (6), (7), (10), (11), (12), (13), (14), (15), (16), (17), and (18)
- (21) Measure volume of sample in separatory funnel using 1 L graduated cylinder and record.
- (22) If there is more than one fraction of extract, combine 100 uL of each fraction into a vial prior to analysis.

### ANALYSIS PROCEDURE:

N/A

### QUALITY CONTROL REQUIREMENTS:



Client:  
SOP ID:  
Rev. Number:  
Rev. Date:

<sup>mz 1-20-97</sup>  
IDEM-BAA-95-38  
SOP-WET-2510-1  
2.0  
March 1, 1996

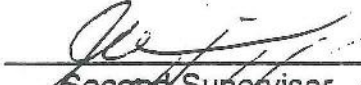
## Standard Operating Procedure For Specific Conductance For Aqueous Samples

Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Wet Chemistry, Main Laboratory

Standard Methods for the Examination of Water and Wastewater, 17th Edition,  
Method 2510 B

Revision # 2.0  
Issued: March 1, 1996

  
\_\_\_\_\_  
Immediate Supervisor      1-20-97  
Date

  
\_\_\_\_\_  
Second Supervisor      1-18-97  
Date

  
\_\_\_\_\_  
QA/QC Officer      1-20-97  
Date

  
\_\_\_\_\_  
Analyst      1-20-97  
Date

Effective: March 1, 1996

### CAUTION

**Disclaimer:** This Standard Operating Procedure has been prepared for the sole use of American Analytical, Inc. and may not be specifically applicable to the activities of other organizations.

Client:	IDEM-BAA 95-30
SOP ID:	SOP-WET-2510-1
Rev. Number:	2.0
Rev. Date:	March 1, 1996

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SOP ID: SOP-WET-2510-1  
Rev. Number 2.0  
Rev. Date: March 1, 1996  
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**STANDARD OPERATING PROCEDURE**  
**SPECIFIC CONDUCTANCE**  
**FOR AQUEOUS SAMPLES**

**LOCATION:**

Wet Chemistry, Main Laboratory

**REFERENCE:**

2510B, Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989

**MATRIX:**

Aqueous, waters, wastewaters, and leachates

**DETECTION LIMIT:**

10 umhos/cm

**RANGE:**

10 to 100,000 umhos/cm

**PRINCIPLE, SCOPE, AND APPLICATION:**

Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions, their total concentration, mobility, valence, relative concentrations, and measurement of temperature. The procedure involves submerging a conductivity probe, equipped with two electrodes, into a sample, adjusting sample temperature to 25°C, and recording the obtained value.

Client: IDEM-3AA 95-30  
SOP ID: SOP-WET-2510-1  
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### INTERFERENCES AND CORRECTIVE ACTION:

Be sure no air bubbles are trapped in the open area of the conductance probe. This may cause false conductance or temperature readings. The trapped air can easily be released by tapping the probe gently once it has been inserted into the sample.

### SAFETY PRECAUTIONS:

Wear a lab coat, safety glasses, and gloves. Glassware is slippery when wet. Handle with care.

### SAMPLE SIZE, COLLECTION, PRESERVATION, AND HANDLING:

Samples (500 ml) should be collected in 500 ml plastic bottles and kept at 4°C until analysis. Holding time is 28 days from collection. No preservation is necessary. Samples will be rejected if bottle is cracked or broken, at which time the immediate supervisor will be notified.

### APPARATUS:

- 1) Conductivity meter (Orion model 122)
- 2) 1" Cuvets compatible with conductivity probe

### ROUTINE MAINTENANCE:

Check batteries in conductivity meter.

### REAGENTS AND CALIBRATION STANDARDS:

- 1) 0.01 M KCl: Dissolve 0.7456 g of anhydrous KCl in reagent water and dilute to 1.0 L at 25°C (0.01 M KCl at 25°C has a conductivity of 1413 umhos/cm)
- 2) Reagent water

### CALIBRATION PROCEDURES:

Check to see that the meter is reading the correct temperature of the sample. Insert the probe into the sample and take the temperature reading and at the same time, insert a thermometer and see if readings match.

### SAMPLE PREPARATION:

Mix sample well to ensure homogeneity

### ANALYSIS PROCEDURE:

- 1) Pour sample into a clean 1" cuvet.
- 2) Place clean probe into cuvet, making sure both electrodes are submerged. Be sure no air bubbles are present on or around the probe.
- 3) Set the meter to the temperature setting. Adjust temperature to 25°C by holding cuvet under lukewarm or cold water.
- 4) Once the temperature reads 25 C, switch the meter setting to read the specific conductance. Read and record observed value.

### QUALITY CONTROL REQUIREMENTS:

See Table I attached for QA/QC procedures.

### CALCULATIONS:

N/A

### DATA DELIVERABLES:

See Table II for data deliverables.

**TABLE I**  
**QUALITY CONTROL LIMITS**

<u>QC Analysis</u>	<u>Frequency</u>	<u>Control Limits umhos/cm</u>
Calibration Curve	6 months	N/A
ICV	1 per batch	1409
CCV	1 per 10 samples	1409
LCS	1 per 10 samples	1409
ICB	1 per batch	<10.0
CCB	1 per 10 samples	<10.0
Preparation Blank	1 per 10 samples	<10.0
Matrix Spike	1 per 10 samples	<10.0
Matrix Duplicate	1 per 10 samples	90 - 110 10% RPD

**TABLE II**  
**DATA DELIVERABLES**

**GENERAL INORGANICS**

**I. PROVIDE DATES AND METHODS AS:**

- 1) Sampling date
- 2) Analysis date and time of day
- 3) Report date
- 4) Analytical, extraction, and digestion methods.
- 5) Extraction date (when applicable)
- 6) Digestion date (when applicable)
- 7) IDEM OSEWM sample number, laboratory number

**II. RECORDER OUTPUTS**

**III. RESULTS OF METHOD AND LAB BLANKS INCLUDING DETECTION LIMITS**

**IV. RESULTS OF STANDARD CALIBRATION INCLUDING:**

- 1) Calibration curve
- 2) Correlation coefficients, and
- 3) standard deviation and relative standard deviation data (when applicable)

**V. CONTINUING CALIBRATION VERIFICATION INCLUDING:**

- 1) Results of mid level standard, and
- 2) Percent recovery

**VI. RESULTS OF MATRIX SPIKE OR MATRIX SPIKE/MATRIX SPIKE DUPLICATE INCLUDING:**

- 1) Amount of spike
- 2) Percent recovery
- 3) Relative percent difference

**VII. RESULTS OF LABORATORY CONTROL SAMPLE**

**VIII. HOLDING TIMES SUMMARY REPORT**

**IX. DETECTION LIMIT SUMMARY REPORT**


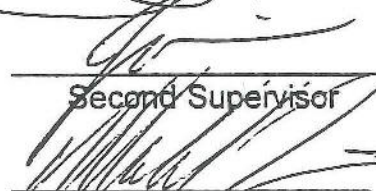
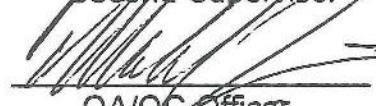
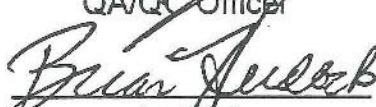
Client: IDEM-BAA 95-30  
SOP ID: SOP-WET-2710F-1  
Rev. Number: 2.0  
Rev. Date: March 1, 1996  
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**Standard Operating Procedure For  
Specific Gravity  
For Aqueous Samples**

Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Wet Chemistry, Main Laboratory

Standard Methods for the Examination of Water and Wastewater, 17th Edition,  
Method 2710 F

Revision # 2.0  
Issued: March 1, 1996

 Immediate Supervisor	1-20-97 Date
 Second Supervisor	1/15/97 Date
 QA/QC Officer	1-20-97 Date
 Analyst	1/20/97 Date

Effective: March 1, 1996

**CAUTION**

**Disclaimer:** This Standard Operating Procedure has been prepared for the sole use of American Analytical, Inc. and may not be specifically applicable to the activities of other organizations.

Client: IDEM-BAA 95-30  
SOP ID: SOP-WET-2710F-1  
Rev. Number: 2.0  
Rev. Date: March 1, 1996  
Page: 1

**STANDARD OPERATING PROCEDURES  
FOR SPECIFIC GRAVITY  
FOR AQUEOUS SAMPLES**

**LOCATION:**

Wet Chemistry, Main Laboratory

**REFERENCE:**

2710 F Standard Methods for the Examination of Water and Wastewater, 17th Edition

**MATRIX:**

Waters, wastewaters, leachates

**DETECTION LIMITS:**

N/A

**RANGE:**

N/A

**PRINCIPLE, SCOPE AND APPLICATION:**

The specific gravity of a sample is the ratio of the masses of equal volume of sample and distilled water. It is determined by comparing the mass of a known volume of sample at a specific temperature to the mass of the same volume of distilled water at 4C.

**INTERFERENCES AND CORRECTIVE ACTION:**

Try to add sample to the container without exerting pressure, or trapping air bubbles as this may cause inaccurate weight measurements.

Client: IDEM-BAA 95-30  
SOP ID: SOP-WET-2710F-1  
Rev. Number: 2.0  
Rev. Date: March 1, 1996  
Page: 2

### SAFETY PRECAUTIONS:

Wear a laboratory coat, safety glasses and gloves at all times.

### SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Sample should be collected in a 1 L plastic container with a teflon lined lid. The sample should be stored in a cooler at 4C until analysis. Sample will be rejected if container is cracked or broken.

### APPARATUS:

- 1) Analytical balance
- 2) 10 mL volumetric flask
- 3) Pasteur pipets and rubber bulbs
- 4) Thermometer (reading up to at least 50 C)

### ROUTINE MAINTENANCE:

The analytical balance is calibrated daily with varying weights and is professionally cleaned once a year.

### REAGENTS AND CALIBRATION STANDARDS:

- 1) DI water at 4'C

### CALIBRATION PROCEDURES:

N/A

### SAMPLE PREPARATION:

N/A

### ANALYSIS PROCEDURE:

- 1) Weigh the clean, dry, and empty 10 mL volumetric flask and record the weight, **W**.
- 2) Fill the 10 mL volumetric flask to the mark with DI water at 4°C, weigh, and record the weight, **R**.
- 3) Empty the volumetric flask and dry completely. Acetone may be used to rinse the inside of the flask. Dry completely by applying a vacuum over the top of the flask to get rid of the excess acetone.
- 4) Fill the 10 mL volumetric flask to the mark with sample, weigh, and record the weight, **S**.
- 5) Record the sample temperature, **T**.

### QUALITY CONTROL:

See Table I for QA/QC procedures.

### DATA TREATMENT:

$$\text{Specific Gravity} = [(S - W) / (R - W)] F$$

where,

F = the temperature correction factor at temperature, T obtained from table below.

<u>Temperature C</u>	<u>Correction</u>
15	0.9991
20	0.9982
25	0.9975
30	0.9957
35	0.9941
40	0.9922
45	0.9903

Client: IDEM-BAA 95-30  
SOP ID: SOP-WET-2710F-1  
Rev. Number: 2.0  
Rev. Date: March 1, 1996  
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DATA DELIVERABLES:

Refer to Table II for data deliverables.

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SOP ID:  
Rev. Number:  
Rev. Date:

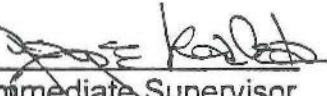

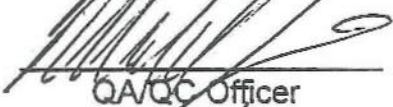
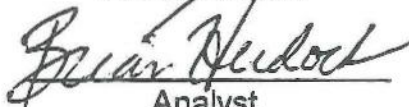
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IDEM-BAA-95-98  
SOP-WET-1601-1  
2.0  
March 1, 1996

## Standard Operating Procedure For Total Dissolved Solids For Aqueous Samples

Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Wet Chemistry, Main Laboratory

EPA 600/4-79-020, Method 160.1

Revision # 2.0  
Issued: March 1, 1996

	1-20-97
Immediate Supervisor	Date
	1/19/97
Second Supervisor	Date
	1-20-97
QA/QC Officer	Date
	1/20/97
Analyst	Date

Effective: March 1, 1996

### CAUTION

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Client:	IDEM-BAA 95-30
SOP ID:	SOP-WET-1601-1
Rev. Number:	2.0
Rev. Date:	March 1, 1996

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**STANDARD OPERATING PROCEDURES**  
**TOTAL DISSOLVED SOLIDS**  
**FOR AQUEOUS SAMPLES**

**LOCATION:**

Wet Chemistry, Main Laboratory

**REFERENCE:**

2540 C - Standard Methods for the Examination of Water & Wastewater 17th Edition. EPA 600/9-79-020, method 160.1, 1971

**MATRIX:**

Water, Wastewater, Leachate, & Aqueous Samples

**DETECTION LIMITS:**

10.0 mg/L

**RANGE:**

10.0 mg/L to 10,000 mg/L

**PRINCIPLE, SCOPE AND APPLICATION:**

A well-mixed sample is filtered through a standard glass fibre filter and the filtrate is evaporated to dryness in a preweighed evaporating dish at 180°C. The increase in dish weight represents the total dissolved solids.

**INTERFERENCES AND CORRECTIVE ACTION:**

Highly mineralized water with a considerable calcium, magnesium, chloride and/or sulfate content may be hygroscopic and require prolonged drying, proper dessication, and rapid weighing. Samples high in bicarbonate require careful and

possibly prolonged drying at 180°C to insure complete conversion of bicarbonate to carbonate.

#### SAFETY PRECAUTIONS:

Wear safety glasses, lab coats and gloves while working with samples. Glassware is very slippery when wet. Be careful when handling to avoid breakage and cuts.

#### SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

No preservation is necessary. Samples (500 ml) may be collected in glass or plastic containers. Samples will be stored at 4 C until analyzed. Holding time is 7 days from collection. Sample will be rejected if container is broken or cracked. At which point the immediate supervisor will be notified.

#### APPARATUS:

- 1) 125 ml Evaporating Dishes (Fisher, 08-710B)
- 2) Dessicator
- 3) Colored Indicator Dessicant (Fisher, 07-758-4A)
- 4) Non-Indicating Dessicant (Fisher, 07-577-4A)
- 5) Drying Oven: 180 C +/- 2 C.
- 6) Buchner Funnel (Fisher, 10-356E. Plate diameter is 114 mm. Perforated area is 95mm).
- 7) 500 mL Vacuum Flask (Fisher, 10-180E)
- 8) Whatman Glass Microfibre Filters. (Fisher, 09-874-16C. Diameter is 110 mm.)
- 9) Analytical Balance to 0.0001 g.

### ROUTINE MAINTENANCE:

Daily calibration of balance with varying weights, yearly professional calibration of balance. Dishes are checked for cracks. Oven is calibrated  $180^{\circ}\text{C} \pm 2^{\circ}\text{C}$  daily.

### REAGENT AND CALIBRATION STANDARDS:

- 1) Reagent water
- 2) 1000 mg/L NaCl: 1.000g NaCl dissolved in DI water and brought up to 1 L in a volumetric flask.

### CALIBRATION PROCEDURES:

Daily calibration of balance with varying weights. Yearly professional calibration of balance. Oven is calibrated at  $108^{\circ}\text{C} \pm 2^{\circ}\text{C}$  daily.

### SAMPLE PREPARATION:

Sample is ready for analysis. Sample is shaken well to mix.

### ANALYSIS PROCEDURE:

- 1) Place filter disc in filtration apparatus and wash three 20 ml portions of reagent water through. Discard washings.
- 2) Heat clean evaporating dish to  $180^{\circ}\text{C} \pm 2^{\circ}\text{C}$  for at least 1 hour in oven. Dessicate at least 1/2 hour to cool.
- 3) Weigh dish immediately before use.
- 4) With vacuum on, filter a measured volume of sample (usually 100 ml) through filter.
- 5) Wash cylinder with three 20 ml portions of reagent water and apply vacuum suction until filter is dry.
- 6) Transfer filtrate to a preweighed evaporating dish.

7) Place dish in oven and dry completely at 180 C +/- 2 C.

8) Cool in a dessicator for at least 1/2 hour and weigh.

QUALITY CONTROL:

See Table I attached for QA/QC procedures.

CALCULATIONS:

$$\text{mg total dissolved solids / L} = \frac{A - B}{\text{Sample vol., L}} \times 1,000$$

Where A = Weight of dish + sample (g)

B = Weight of dish (g)

DATA DELIVERABLE:

See Table II for data deliverables.

**TABLE I**  
**QUALITY CONTROL LIMITS**

<u>QC Analysis</u>	<u>Frequency</u>	<u>Control Limits % Recovery</u>
Calibration Curve	6 months	N/A
ICV	1 per batch	N/A
CCV	1 per 10 samples	N/A
LCS	1 per 10 samples	N/A
ICB	1 per batch	<10.0
CCB	1 per 10 samples	<10.0
Preparation Blank	1 per 10 samples	<10.0
Matrix Spike	1 per 10 samples	<10.0
Matrix Duplicate	1 per 10 samples	80 - 120 20% RPD

**TABLE II**  
**DATA DELIVERABLES**

**GENERAL INORGANICS**

**I. PROVIDE DATES AND METHODS AS:**

- 1) Sampling date
- 2) Analysis date and time of day
- 3) Report date
- 4) Analytical, extraction, and digestion methods.
- 5) Extraction date (when applicable)
- 6) Digestion date (when applicable)
- 7) IDEM OSEWM sample number, laboratory number

**II. RECORDER OUTPUTS**

**III. RESULTS OF METHOD AND LAB BLANKS INCLUDING DETECTION LIMITS**

**IV. RESULTS OF STANDARD CALIBRATION INCLUDING:**

- 1) Calibration curve
- 2) Correlation coefficients, and
- 3) standard deviation and relative standard deviation data (when applicable)

**V. CONTINUING CALIBRATION VERIFICATION INCLUDING:**

- 1) Results of mid level standard, and
- 2) Percent recovery

**VI. RESULTS OF MATRIX SPIKE OR MATRIX SPIKE/MATRIX SPIKE DUPLICATE INCLUDING:**

- 1) Amount of spike
- 2) Percent recovery
- 3) Relative percent difference

**VII. RESULTS OF LABORATORY CONTROL SAMPLE**

**VIII. HOLDING TIMES SUMMARY REPORT**

**IX. DETECTION LIMIT SUMMARY REPORT**

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SOP ID:  
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M21-20-97  
IDEM-BAA-95-36  
SOP-ORG-9060-1  
1.0  
October 14, 1996  
1

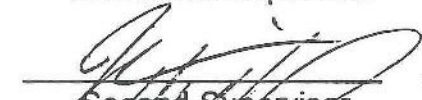
## Standard Operating Procedure For Total Organic Carbon (TOC) For Aqueous Samples


Author: Karin Stewart  
Prepared For American Analytical, Inc.  
Organics, Organics Laboratory & Main Laboratory

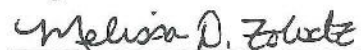
SW-846, 3rd Edition, Method 9060

Revision # 1.0  
Issued: October 14, 1996

  
\_\_\_\_\_  
Immediate Supervisor      1/17/97  
Date

  
\_\_\_\_\_  
Second Supervisor      1/17/97  
Date

  
\_\_\_\_\_  
QA/QC Officer      1-17-97  
Date

  
\_\_\_\_\_  
Analyst      1/17/97  
Date

Effective: October 14, 1996

### CAUTION

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Client:	IDEM-BAA 95-30
SOP ID:	SOP-ORG-9060-1
Rev. Number:	1.0
Rev. Date:	October 14, 1996
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Client: IDEM BAA 96-30  
SOP ID: SOP-ORG-9060-1  
Rev Number: 1.0  
Rev Date: October 14, 1996  
Page: 1

**STANDARD OPERATING PROCEDURE**  
**TOTAL ORGANIC CARBON (TOC)**  
**AQUEOUS SAMPLES**

**LOCATION:**

Metals Laboratory

**REFERENCE:**

SW-846, 3rd Edition, Method 9060

**MATRIX:**

Water, Leachate

**DETECTION LIMITS:**

1.00 mg/l

**RANGE:**

1.00 mg/l - 150 mg/l

**PRINCIPAL, SCOPE, AND APPLICATION:**

This method is used to determine the concentration of organic carbon in ground water, surface and saline waters, and domestic and industrial wastewater.

Organic carbon is measured using a carbonaceous analyzer. This instrument converts the organic carbon in the sample to carbon dioxide by chemical oxidation. The carbon dioxide is then measured directly by an infrared detector. The amount of carbon dioxide in a sample is directly proportional to the concentration of carbonaceous material in the sample.

Carbonaceous analyzers are capable of measuring all forms of carbon in a sample. However, because of various properties of carbon-containing compounds in liquid samples, the manner of

Client: IDEM BAA 96-30  
SOP ID: SOP-ORG-9060-1  
Rev Number: 1.0  
Rev Date: October 14, 1996  
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preliminary sample treatment as well as instrument settings will determine which forms of carbon are actually measured. The forms of carbon that can be measured by this method:

1. Soluble, nonvolatile organic carbon: e.g., natural sugars.
2. Soluble, volatile organic carbon: e.g., meraptans, alkanes, low molecular weight alcohols.
3. Insoluble, partially volatile carbon: e.g., low molecular weight oils.
4. Insoluble, particulate carbonaceous materials: e.g. cellulose fibers.
5. Soluble or insoluble carbonaceous materials adsorbed or entrapped on insoluble inorganic suspended matter: e.g., oily matter adsorbed on silt particles.

Carbonate and bicarbonate are inorganic forms of carbon and must be separated from the total organic carbon value. This is accomplished by converting the carbonate and bicarbonate to carbon dioxide and degassing prior to analysis.

#### INTERFERENCES AND CORRECTIVE ACTION:

Carbonate and bicarbonate carbon represent an interference under the terms of this test and must be accounted for in the final calculation.

This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microliter-type syringe or pipet. The openings of the syringe or pipet limit the maximum size of particle which may be included in the sample.

Removal of carbonate and bicarbonate by acidification and purging with nitrogen, or other inert gas, can result in the loss of volatile organic substances.

Chloride ion can have a profound effect on the oxidation rate of organics in the UV-persulfate system. Normally chloride levels of about 0.10% will be the starting point where the effect is noticeable. If chloride is present or baseline problems exists dilute the sample to reduce the chloride ion concentration.

#### SAFETY PRECAUTIONS:

Lab coats and safety glasses are to be worn at all times.

Client: IDEM BAA 96-30  
SOP ID: SOP-ORG-9060-1  
Rev Number: 1.0  
Rev Date: October 14, 1996  
Page: 3

SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING:

Samples are to be collected in glass 250ml containers with teflon lined lids, preserved to pH < 2 with H<sub>2</sub>SO<sub>4</sub> and kept cool (4C). Samples must be analyzed within 28 days.

APPARATUS AND MATERIALS:

- 1) Dohrmann DC-80 Total Organic Carbon Analyzer.
- 2) Microliter type syringe, 1.0ml
- 3) Class A volumetric flasks
- 4) Class A volumetric pipets

ROUTINE MAINTENANCE:

Check gas daily and change when low. Inspect tubing and change as needed.

REAGENTS AND CALIBRATION STANDARDS:

- 1) Reagent water: ASTM Type II water boiled and cooled to remove CO<sub>2</sub>.
- 2) Potassium Persulfate, 2% solution: Dissolve 20 grams of Potassium Persulfate in 900ml of reagent water add 1ml of concentrated phosphoric acid and dilute to 1 liter.
- 3) Organic Carbon Standard, 2000 ppm Stock Solution: Dissolve .425 grams of dried Potassium Hydrogen Phthalate in 90ml reagent water, add 0.1 ml of concentrated phosphoric acid, and dilute to 100ml. Store in amber glass container and refrigerate.
- 4) 150 ppm C standard solution: Add 7.5ml of 2000 ppm stock standard solution to 100ml volumetric flask and dilute to mark with reagent water. Store in amber glass container and refrigerate.
- 5) 10.0 ppm C standard solution: Add 0.5ml of 2000 ppm stock standard solution to a 100ml volumetric flask and dilute to mark with reagent water. Store in amber glass container and refrigerate.
- 6) 2.0 ppm C standard solution: Add 0.1ml of 2000 ppm stock standard solution to a 100ml volumetric flask and dilute to

Client: IDEM BAA 96-30  
SOP ID: SOP-ORG-9060-1  
Rev Number: 1.0  
Rev Date: October 14, 1996  
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mark with reagent water. Store in amber glass container and refrigerate.

7) Blank solution: Same type II water used to make standards.

#### START-UP PROCEDURES:

1) Turn on white power switches for the main power, pump, lamp, and electronics.

2) Open the gas cylinder and adjust outlet pressure to 30 psig. Measure the gas flow at the detector out port to verify that the flow is in the range of 180-220 ml/min.

3) Verify that the reactor is filled with reagent, printer tape is installed, mode switch is in the TOC position, and the detector switch is in the ppm position.

4) After approximately 10 min warm up and if the detector has been on for two hours or more the instrument is ready to operate. The detector generally should be left on at all times, except when used intermittently, and then it should be turned on the day before use.

5) Clear the calibration factor memory by pressing the "CALIB" button for at least a full second, the light will turn off.

6) Place approximately 15ml of the 10ppm standard in a testube and acidify to  $\text{pH} < 2$  with five drops of 10% phosphoric acid and sparge for at least three minutes.

7) Set volume select switch to 1ml. Press the green start button and inject 1ml of the 10 ppm standard. The ready light will go out and the start light will come on.

8) The display will show the intergrated signal as a four digit number. Lift the protective cap on the yellow "CALIB" switch. The yellow light will come on.

9) The signal generated by the detector is fed to the microprocessor and it calculates the peak area above the baseline. The peak is multiplied by an arbitrary constant which has previously stored, to obtain a more reasonable number ( $a_1$ ) which is close to the ppm of the standard. This number is displayed on the meter and printed on the tape at the end of an analysis.

10) The microprocessor compares  $a_1$  with the value preset at the factory to determine whether or not  $a_1$  is a reasonable

Client: IDEM BAA 96-30  
SOP ID: SOP-ORG-9060-1  
Rev Number: 1.0  
Rev Date: October 14, 1996  
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value. If it is not, the message is printed on the tape, like ABNORMAL HIGH STD.

11) If this is the case stop analysis and check tubing and connections for leaks. Also make sure the correct standard solution was injected.

#### CALIBRATION PROCEDURES:

1) Place approximately 15ml of blank in a testtube, acidify with five drops of 10% phosphoric acid and sparge for at least three minutes.

2) Withdraw 1ml, push green start button and inject. The ready light will go out and the start light will come on.

3) The digital display will show increasing accumulation of counts. When the detector signal is essentially back to original base line, the analysis will terminate automatically.

4) The ready light will come on and the start light will go off. The display will show the integrated signal as a four digit number and it will also be printed.

5) Repeat steps 1 through 4 with the 2.0ppm, 10ppm, 150ppm standards. Input concentration vs. instrument output into calculator capable of performing linear regression analysis. Correlation coefficient must be  $\geq 0.995$ , if not terminate and begin from the start.

#### SAMPLE PREPARATION AND ANALYSIS PROCEDURE:

1) Place approximately 15ml of samples in a testtube, acidify to pH 2-3 with five drops of 10% phosphoric acid and sparge for at least five minutes.

2) Withdraw 1.0ml of sample, push green start button and inject.

3) The digital display will show increasing accumulation of counts. When the detector signal is essentially back to original base line, the analysis will terminate automatically.

4) The ready light will come on and the start light will go off. The display will show the integrated signal as a four digit number and it will also be printed.

5) Input the result into the calculator. The linear regression will convert the instrument output into concentration of carbon in ppm.

Client: IDEM BAA 96-30  
SOP ID: SOP-ORG-9060-1  
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#### QUALITY CONTROL:

- 1) This method requires all samples be run in duplicate.
- 2) Dilute and reanalyze samples that are more concentrated than the high standard.
- 3) Analyze a matrix spike and matrix spike duplicate every ten samples. The RPD must be within 20%.
- 4) Verify calibration with a midlevel standard every ten samples. The check standard should agree within 10%. If it does not, analysis will be terminated and instrument recalibrated.
- 5) Recalibrate every twenty samples.

#### DATA TREATMENT:

The concentration of Organic Carbon in ppm is calculated from the linear regression (calibration curve).

If dilution was required:

$$\text{ppm or mg/l Organic Carbon in sample} = A \times \frac{(C+B)}{C}$$

where,

A = mg/l of Organic Carbon from calibration curve

B = blank used for dilution, ml

C = sample aliquot, ml

Relative percent difference is calculated as follows:

$$\text{RPD} = \frac{D_1 - D_2}{(D_1 + D_2) / 2} \times 100$$

RPD = Relative Percent Difference

D<sub>1</sub> = First sample value

D<sub>2</sub> = Second sample value

The spike recovery is calculated as follows:

$$\%R = \frac{SS - S}{SA} \times 100$$

%R = Percent recovered

SS = Spike sample value

S = Sample value

Client: IDEM BAA 96-30  
SOP ID: SOP-ORG-9060-1  
Rev Number: 1.0  
Rev Date: October 14, 1996  
Page: 7

SA = Spike Added

DATA DELIVERABLES:

Reports to client will include:

- Date of receipt
- Date of preparation
- Date of analysis
- Analyst
- Matrix
- Laboratory ID#
- Client ID#
- Analytical method#
- Concentrations determined and EQL's
- CCV summary form
- Spike sample recovery form
- Duplicate spike sample summary form
- All raw data
- Preparation records

## APPENDIX D

## ATTACHMENT #1

June 12, 1997  
#5a:\J021uic.doc

**PLUGGING AND ABANDONMENT COST ESTIMATE**

**Subject: Burns Harbor Division  
Underground Injection Wells**

1. Annual (Average) Producer Price Index for Oil and Gas Field Machinery (Commodity Code 11-91) from U.S. Department of Labor (202-606-7705) (values are also available off the Internet):

1995 - 114.1

1996 - 117.8

2. Inflation Factor        =    1996 Index/1995 Index  
    (1997)                =    117.8/114.1  
                             =    1.032

3. 1997 plugging and abandonment cost estimate is calculated by adjusting the 1996 cost estimates for inflation based on the above price index:

WPL #1	\$151,500	x	1.032	=	156,400
WAL #1	\$ 88,000	x	1.032	=	90,800
WAL #2	\$ 88,000	x	1.032	=	90,800
GSMW*	\$ 81,100	x	1.032	=	83,700
<b>TOTAL</b>					<b>\$421,700</b>

\* GSMW = Galesville Sandstone Monitoring Well

4. Attachment #2 is a table showing the history of the cost estimates from 1986 to 1997.

*D. L. Holmes*

D. L. Holmes

<b>BURNS HARBOR DIVISION</b> <b>UNDERGROUND INJECTION CONTROL (UIC) WELLS</b> <b>PLUGGING AND ABANDONMENT COST ESTIMATES - \$</b>						
YEAR	INFLATION FACTOR	WPL #1	WAL #1	WAL #2	GSMW	TOTAL
1986	*	46,500	36,200	36,200	--	118,900
1987	0.973	45,300	35,200	35,200	--	115,700
1988	0.989	44,800	34,850	34,850	--	114,500
1989	1.041	46,600	36,300	36,300	--	119,200
1990	**	131,000	76,000	76,000	70,000	353,000
1991	1.033	135,500	78,500	78,500	72,500	365,000
1992	1.060	144,000	83,000	83,000	77,000	387,000
1993	0.991	142,500	82,500	82,500	76,000	383,500
1994	1.006	143,400	83,300	83,300	76,700	386,700
1995	1.024	146,900	85,300	85,300	78,600	396,100
1996	1.031	151,500	88,000	88,000	81,100	408,600
1997	1.032	156,400	90,800	90,800	83,700	421,700
* Initial cost estimate. ** Revised cost estimate. WPL = Waste Pickle Liquor WAL = Weak Ammonia Liquor GSMW = Galesville Sandstone Monitoring Well						

**INFLATION FACTORS  
UNDERGROUND INJECTION CONTROL (UIC) WELLS  
PLUGGING AND ABANDONMENT COST ESTIMATES \$**

<b>YEAR</b>	<b>Annual Producer Price Index for Oil and Gas Field Machinery (Commodity Code 11-91)***</b>	<b>INFLATION FACTOR</b>
1985	426.2	--
1986	414.9/94.3*	
1987	93.3	$414.9/426.2 = 0.973$
1988	97.1	$93.3/4.39 = 0.989$
1989	99.1	$97.1/93.3 = 1.041$
1990	102.4	$99.1/97.1 = 1.021$
1991	108.6	$102.4/99.1 = 1.033$
1992	107.6	$108.6/102.4 = 1.060$
1993	108.2	$107.6/108.6 = 0.991$
1994	110.8	$108.2/107.6 = 1.006$
1995	114.2	$110.8/108.2 = 1.024$
1996	117.8	$114.2/110.8 = 1.031$
1997	--	$117.8/114.1^{**} = 1.032$

\* U.S. Department of Labor converted to a new base.  
Phone Number: (202) 606-7705 (valid as of 6/12/97)

\*\* 1995 value changed from 114.2 to 114.1 in 1997.

\*\*\* Available on Internet @ <http://stats.bls.gov:80/cgi-bin/srgate>

# Bureau of Labor Statistics Data

Data extracted on: June 11, 1997 (04:34 PM)

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## Producer Price Index-Commodities

### Series Catalog:

Series ID : WPU1191

Not Seasonally Adjusted

Group : Machinery and equipment

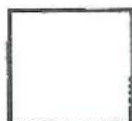
Item : Oil field and gas field machinery

Base Date : 8200

Data:

Year	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Ann
1987	93.4	93.2	93.3	92.8	92.8	92.5	92.3	92.7	92.9	93.5	94.8	95.3	93.3
1988	95.9	95.9	96.2	96.4	97.1	97.1	97.4	97.5	97.8	97.6	97.3	97.8	97.0
1989	97.9	97.9	98.1	98.2	98.5	98.6	99.3	100.1	100.2	100.5	100.2	100.1	99.1
1990	100.7	100.5	100.6	100.7	100.6	100.9	101.2	102.0	104.7	104.9	105.8	106.1	102.4
1991	106.2	106.8	107.8	107.9	109.0	109.0	109.4	109.3	109.3	109.3	109.3	109.3	108.6
1992	109.3	107.6	107.6	107.6	107.7	107.9	107.3	106.8	107.3	106.9	107.9	108.0	107.6
1993	108.2	108.1	108.4	107.6	108.1	107.5	107.3	107.3	108.0	108.9	109.0	109.8	108.2
1994	109.8	110.6	110.4	110.4	110.0	110.2	110.6	110.5	110.8	111.9	111.9	111.9	110.8
1995	112.4	112.6	113.2	113.3	113.3	113.6	114.5	114.6	114.7	115.6	115.8	115.8	114.1
1996	116.9	117.0	117.0	117.1	117.1	117.8	118.0	118.0	118.1	118.5	118.8	119.4	117.8
1997	121.0(P)	121.0(P)	121.7(P)	122.1(P)									

P : Preliminary. All indexes are subject to revision four months after original publication.



[Data Home Page](#)



[BLS Home Page](#)

Bureau of Labor Statistics  
[catron\\_b@bls.gov](mailto:catron_b@bls.gov)

# Bethlehem Steel Corporation

1170 Eighth Avenue  
BETHLEHEM, PA 18016-7699

H. ROGER FARBER  
DIRECTOR, RISK MANAGEMENT  
FINANCE DEPARTMENT



PHONE (610) 694-7463  
FAX (610) 694-3356

June 26, 1997

Via Federal Express

Ms. Rebecca Harvey, Branch Chief  
U. S. Environmental Protection Agency  
Region 5  
77 West Jackson Boulevard  
Chicago, IL 60604-3507

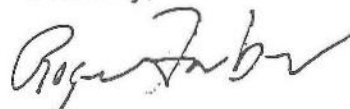
Attn: MS(WU-17J)

Dear Ms. Harvey:

In accordance with 40 CFR Part 144, Bethlehem previously provided a fully-funded Trust Agreement to meet our financial responsibility obligations for the UIC wells at our Burns Harbor Division. Enclosed is a Surety Performance Bond and a new Standby Trust Agreement which are being submitted to replace the existing Trust Agreement in a dollar-for-dollar substitution. Please review, acknowledge acceptance, and grant your release of the existing funded Trust Agreement.

If you have any questions, please contact me at (610) 694-7463.

Sincerely,



H. Roger Farber

Enclosures

# SURETY PERFORMANCE BOND

U.S. Environmental Protection Agency,  
Underground Injection Control  
Financial Responsibility Requirement

BOND COVERS THE PLUGGING OF INJECTION WELLS

Date bond executed: June 30, 1997

Effective date: June 30, 1997

Principal: Bethlehem Steel Corporation  
(Legal name of owner or operator)

Bethlehem, PA 18016-7699  
(Business address of owner or operator)

Type of organization: Corporation  
(Individual, joint venture,  
partnership, or corporation)

State of incorporation: Delaware

Surety(is): Frontier Insurance Company  
(Name)

195 Lake Louise Marie Road, Rock Hill, NY 12775-8000  
(Business Address)

EPA identification number, name, address, and plugging and abandonment amount(s) for each injection well guaranteed by this bond. (Indicate plugging and abandonment amounts for each well. Attach separate list if necessary.)

Injection Well Information	Plugging & Abandonment Amount
WPL No. 1 - EPA ID No. IN-127-1W-0001	\$ 156,400
WAL No. 1 - IN-127-1W-0003	90,800
WAL No. 2 - IN-127-1W-0004	90,800
GSMW - Not Applicable	83,700
Burns Harbor Division, Bethlehem Steel Corp. U.S. Route 12, Westchester Township, IN 46304	

Total penal sum of bond: \$ 421,700

Surety's bond number: 108364

KNOW ALL PERSONS BY THESE PRESENTS, That we, the Principal and Surety(ies) hereto are firmly bound to the U.S. Environmental Protection Agency (hereinafter called EPA), in the above penal sum for the payment of which we bind ourselves, our heirs, executors, administrators, successors, and assigns jointly and severally; provided that, were the Surety(ies) are corporations acting as co-sureties, we, the Sureties, bind ourselves in such sum "jointly and severally" only for the purpose of allowing a joint action or actions against any or all of us, and for all other purposes each Surety binds itself, jointly and severally with the Principal, for the payment of such sum only as is set forth opposite the name of such surety, but if no limit of liability is indicated, the limit of liability shall be the full amount of the penal sum.

WHEREAS said Principal is required, under the Underground Injection Control Regulations, as amended, to have a permit or comply with provisions to operate under rule for each injection well identified above, and

WHEREAS said Principal is required to provide financial assurance for plugging and abandonment as a condition of the permit or approval to operate under rule, and

WHEREAS said Principal shall establish a standby trust fund as is required when a surety bond is used to provide such financial assurance;

NOW, THEREFORE, the conditions of this obligation are such that if the Principal shall faithfully perform plugging and abandonment, whenever required to do so, of each injection well for which this bond guarantees plugging and abandonment, in accordance with the plugging and abandonment plan and other requirements of the permit or provisions for operating under rule and other requirements of the permit or provisions for operating under rule as may be amended, pursuant to all applicable laws, statutes, rules and regulations, as such laws, statutes, rules, and regulations may be amended,

Or, if the Principal shall provide alternate financial assurance as specified in Subpart F of 40 CFR 144, and obtain the EPA Regional Administrator's written approval of such assurance, within 90 days after the date of notice of cancellation is received by both the Principal and the EPA Regional Administrator(s) from the Surety(ies), then this obligation shall be null and void. Otherwise it is to remain in full force and effect.

The Surety(ies) shall become liable on this bond obligation only when the Principal has failed to fulfill the conditions described above.

Upon notification by an EPA Regional Administrator that the Principal has been found in violation of the plugging and abandonment requirements of 40 CFR 144, for an injection well which this bond guarantees performances of plugging and abandonment, the Surety(ies) shall either perform plugging and abandonment in accordance with the plugging and abandonment plan and other permit requirements or provisions for operating under rule

and other requirements or place the amount for plugging and abandonment into standby trust fund as directed by the EPA Regional Administrator.

Upon notification by an EPA Regional Administrator that the Principal has failed to provide alternate financial assurance as specified in Subpart F of 40 CFR 144, and obtain written approval of such assurance from the EPA Regional Administrator(s) during the 90 days following receipt by both the Principal and the EPA Regional Administrator(s) of a notice of cancellation of the bond, the Surety(ies) shall place funds in the amount guaranteed for the injection well(s) into the standby trust fund as directed by the EPA Regional Administrator.

- The Surety(ies) hereby waive(s) notification of amendments to plugging and abandonment plans, permits, applicable laws, statutes, rules, and regulations and agrees that no such amendment shall in any way alleviate its (their) obligation on this bond.

The liability of the Surety(is) shall not be discharged by any payment or succession of payments hereunder, unless and until such payment or payments shall amount in the aggregate to the penal sum of the bond, but in no event shall the obligation of the Surety(ies) here under exceed the amount of said penal sum.

The Surety(ies) may cancel the bond by sending notice by certified mail to the owner or operator and to the EPA Regional Administrator(s) for the Region(s) in which the injection well(s) is (are) located, provided, however, that cancellation shall not occur during the 120 days beginning on the date of receipt of the notice of cancellation by both the Principal and the EPA Regional Administrator(s), as evidenced by the return receipts.

The Principal may terminate this bond by sending written notice to the Surety(ies); provided, however, that no such notice shall become effective until the Surety(ies) receive(s) written authorization for termination of the bond by the EPA Regional Administrator(s) of the EPA Region(s) in which the bonded injection well(s) is (are) located.

(The following paragraph is an optional rider that may be included but is not required.)

Principal and Surety(ies) hereby agree to adjust the penal sum of the bond yearly so that it guarantees a new plugging and abandonment amount, provided that the penal sum does not increase by more than 20% in any one year, and no decrease in the penal sum takes place without the written permission of the EPA Regional Administrator(s).

In WITNESS WHEREOF, The Principal and Surety(ies) have executed this Performance Bond and have affixed their seals on the date set forth above.

The persons whose signature appear below hereby certify that they are authorized to execute this surety bond on behalf of the Principal and Surety(ies) and that the wording on this surety bond is identical to the wording specified in 40 CFR 144.70(c) as such regulation was constituted on the date this bond was executed.

## PRINCIPAL:

Bethlehem Steel Corporation

(Name)

Bethlehem, PA 18016-7699

(Address)

A.E. Moffitt, Jr.

(Signature(s))

A.E. Moffitt, Jr.

(Name(s))

Vice President - Safety, Health and Environment

(Title(s))

Corporate Seal

Delaware

State of Incorporation

\$ 3,163.00

Bond Premium

## CORPORATE SURETY(IES):

FRONTIER INSURANCE COMPANY

(Name)

195 Lake Louise Marie Road  
Rock Hill, NY 12775-8000

(Address)

Stephen T. Kazmer

(Signature(s))

Stephen T. Kazmer

(Name(s))

Attorney-in-Fact

(Title(s))

Corporate Seal

New York

State of Incorporation

\$ 421,700.00

Liability Limit

(For every co-surety, provide signature(s), corporate seal, and other information in the same manner as for Surety above.)

State of Illinois  
County of Cook } ss:

On June 30, 1997  
therein, duly commissioned and sworn, personally appeared

, before me, a Notary Public in and for said County and State, residing  
Stephen T. Kazmer

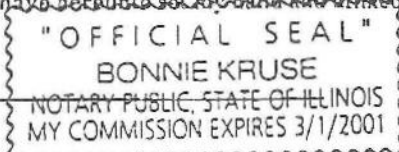
known to me to be Attorney-in-Fact of

Frontier Insurance Company

the corporation described in and that executed the within and foregoing instrument, and known to me to be the person who executed the  
said instrument in behalf of the said corporation, and he duly acknowledged to me that such corporation executed the same.

IN WITNESS WHEREOF, I have hereunto set my hand and affixed my official seal, the day and year stated in this certificate above.

My Commission Expires



*Bonnie Kruse*  
Notary Public

## POWER OF ATTORNEY

Know All Men By These Presents: That FRONTIER INSURANCE COMPANY, a New York Corporation, having its principal office in Rock Hill, New York, pursuant to the following resolution, adopted by the Board of Directors of the Corporation on the 4th day of November, 1985:

"RESOLVED, that the Chairman of the Board, the President, or any Vice President be, and hereby is, authorized to appoint Attorneys-in-Fact to represent and act for and on behalf of the Company to execute bonds, undertakings, recognizances and other contracts of indemnity and writings obligatory in the nature thereof, and to attach thereto the corporate seal of the Company, in the transaction of its surety business;

"RESOLVED, that the signatures and attestations of such officers and the seal of the Company may be affixed to any such Power of Attorney or to any certificate relating thereto by facsimile, and any such Power of Attorney or certificate bearing such facsimile signatures or facsimile seal shall be valid and binding upon the Company when so affixed with respect to any bond, undertaking, recognizance or other contract of indemnity or writing obligatory in the nature thereof;

"RESOLVED, that any such Attorney-in-Fact delivering a secretarial certification that the foregoing resolutions still be in effect may insert in such certification the date thereof, said date to be not later than the date of delivery thereof by such Attorney-in-Fact."

This Power of Attorney is signed and sealed in facsimile under and by the authority of the above Resolution.

DOES HEREBY MAKE, CONSTITUTE AND APPOINT:

Lewis James Scheer Michael J. Scheer Alice Rhoads  
James I. Moore Bonnie Kruse Stephen T. Kazmer Dawn L. Morgan

of La Grange in the State of Illinois  
its true and lawful Attorney(s)-in-Fact with full power and authority hereby conferred in its name, place and stead to sign, execute, acknowledge and deliver in its behalf, and as its act and deed, without power of redelegation, as follows:

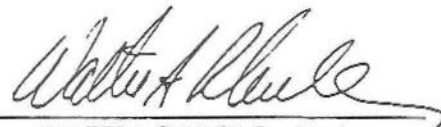
Bonds guaranteeing the fidelity of persons holding places of public or private trust; guaranteeing the performance of contracts other than insurance policies; and executing or guaranteeing bonds and undertakings required or permitted in all actions or proceedings or by law allowed; IN AN AMOUNT NOT TO EXCEED THREE MILLION FIVE HUNDRED THOUSAND (\$3,500,000.00) DOLLARS; and to bind FRONTIER INSURANCE COMPANY thereby as fully and to the same extent as if such bond or undertaking was signed by the duly authorized officers of FRONTIER INSURANCE COMPANY, and all the acts of said Attorney(s)-in-Fact pursuant to the authority herein given are hereby ratified and confirmed.

In Witness Whereof, FRONTIER INSURANCE COMPANY of Rock Hill, New York, has caused this Power of Attorney to be signed by its President and its Corporate seal to be affixed this 18th day of June, 19 90.

FRONTIER INSURANCE COMPANY

State of New York  
County of Sullivan ss.:

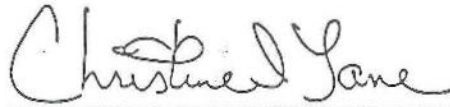


BY:   
WALTER A. RHULEN, President

On this 18th day of June, 19 90, before the subscriber, a Notary Public of the State of New York in and for the County of Sullivan, duly commissioned and qualified, came WALTER A. RHULEN of FRONTIER INSURANCE COMPANY to me personally known to be the individual and officer described herein, and who executed the preceding instrument, and acknowledged the execution of the same, and being by me duly sworn, deposed and said, that he is the officer of the Company aforesaid, and that the seal affixed to the preceding instrument is the Corporate Seal of the Company, and the Corporate Seal and signature as an officer were duly affixed and subscribed to the said instrument by the authority and direction of the Corporation, and that the resolution of the Company, referred to in the preceding instrument, is now in force.

In Testimony Whereof, I have hereunto set my hand, and affixed my official seal at Rock Hill, New York, the day and year above written.





CHRISTINE I. LANE  
Notary Public State of New York  
Sullivan County Clerk's No. 1996  
Commission Expires May 2, 1998

## CERTIFICATION

I, JOSEPH P. LOUGHLIN, Secretary of FRONTIER INSURANCE COMPANY of Rock Hill, New York, do hereby certify that the foregoing Resolution adopted by the Board of Directors of this Corporation and the Powers of Attorney issued pursuant thereto, are true and correct, and that both the Resolution and the Powers of Attorney are in full force and effect.

In Witness Whereof, I have hereunto set my hand and affixed the facsimile seal of the corporation this 30th day of June, 19 97.



  
JOSEPH P. LOUGHLIN, Secretary

For Sample Use Only  
Comparable Wording Acceptable

# STANDBY TRUST AGREEMENT

U.S. Environmental Protection Agency  
Underground Injection Control  
Financial Responsibility Requirement

TRUST AGREEMENT, the "Agreement," entered into as of June 26<sup>th</sup>, 1997  
(date)

by and between Bethlehem Steel Corporation  
(name of owner or operator)

a Delaware Corporation, the "Grantor,"  
(name of state) (corporation, partnership,  
association, or proprietorship)

and Core States Bank N.A., (X) incorporated in the  
(name of corporate trustee)

State of PA or ( ) a national bank, the "Trustee."

WHEREAS, the United States Environmental Protection Agency, "EPA," an agency of the United States Government, has established certain regulations applicable to the Grantor, requiring that an owner or operator of an injection well shall provide assurance that funds will be available when needed for plugging and abandonment of the injection well, and

WHEREAS, the Grantor has elected to obtain (X) a surety bond ( ) a letter of credit and establish a standby trust to provide all or part of such financial assurance for the facility(ies) identified herein, and

WHEREAS, the Grantor, acting through its duly authorized officers, has selected the Trustee to be the trustee under this Agreement, and the Trustee is willing to act as trustee,

NOW, THEREFORE, the Grantor and the Trustee agree as follows:

Section 1. Definitions. As used in this Agreement:

(a) The term "Grantor" means the owner or operator who enters into this Agreement and any successors or assigns of the Grantor.

(b) The term "Trustee" means the Trustee who enters into this Agreement and any successor Trustee.

(c) "Facility" or "activity" means any underground injection well or any other facility or activity that is subject to regulation under the Underground Injection Control Program.

Section 2. Identification of Facilities and Cost Estimates. This Agreement pertains to the facilities and cost estimates identified in Schedule A (attached). (Schedule A lists, for each facility, the EPA identification number, name, address, and the current plugging and abandonment cost estimate, or portions thereof, for which financial assurance is demonstrated.)

Section 3. Establishment of Fund. The Grantor and the Trustee hereby establish a trust fund, the "Fund," for the benefit of EPA. The Grantor and the Trustee intend that no third party have access to the Fund except as herein provided. The Fund is established initially as consisting of the property, which is acceptable to the Trustee, described in Schedule B attached hereto. Such property and any other property subsequently transferred to the Trustee is referred to as the Fund, together with all earnings and profits thereon, less any payments or distributions made by the Trustee pursuant to this Agreement. The Fund shall be held by the Trustee, IN TRUST, as hereinafter provided. The Trustee shall not be responsible nor shall it undertake any responsibility for the amount or adequacy of, nor any duty to collect from the Grantor, any payments necessary to discharge any liabilities of the Grantor established by EPA.

Section 4. Payment for Plugging and Abandonment. The Trustee shall make payments from the Fund as the EPA Regional Administrator shall direct, in writing, to provide for the payment of the costs of plugging and abandonment of the injection wells covered by this Agreement. The Trustee shall reimburse the Grantor or other persons as specified by the EPA Regional Administrator from the Fund for plugging and abandonment expenditures in such amounts as the EPA Regional Administrator shall direct in writing. In addition, the Trustee shall refund to the Grantor such amounts as the EPA Regional Administrator specifies in writing. Upon refund, such funds shall no longer constitute part of the Fund as defined herein.

Section 5. Payments Comprising the Fund. Payments made to the Trustee for the Fund shall consist of cash or securities acceptable to the Trustee.

Section 6. Trustee Management. The Trustee shall invest and reinvest the principal and income of the Fund and keep the Fund invested as a single fund, without distinction between principal and income, in accordance with general investment policies and guidelines which the Grantor may communicate in writing to the Trustee from time to time, subject, however, to the provisions of this Section. In investing, reinvesting, exchanging, selling, and managing the Fund, the Trustee shall discharge his duties with respect to the trust fund solely in the interest of the beneficiary and with the care, skill, prudence, and diligence under the circumstances then prevailing, which persons of prudence, acting in a like capacity and familiar with such matters, would use in the conduct of an enterprise of a like character and with like aims, except that:

(a) Securities or other obligations of the Grantor, or any other owner or operator of the facilities, or any of their affiliates as defined in the Investment Company Act of 1940, as amended, 15 USC 80a-2.(a), shall not be acquired or held, unless they are securities or other obligations of the Federal or a State government;

(b) The Trustee is authorized to invest the Fund in time or demand deposits of the Trustee, to the extent insured by an agency of the Federal or State government; and

(c) The Trustee is authorized to hold cash awaiting investment or distribution uninvested for a reasonable time and without liability for the payment of interest thereon.

Section 7. Commingling and Investment. The Trustee is expressly authorized in its discretion:

(a) To transfer from time to time any or all of the assets of the Fund to any common, commingled, or collective trust fund created by the Trustee in which the Fund is eligible to participate, subject to all of the provisions thereof, to be commingled with the assets of other trusts participating therein; and

(b). To purchase shares in any investment company registered under the Investment Company Act of 1940, 15 U.S.C. 80a-1 et seq., including one which may be created, managed, underwritten, or to which investment advice is rendered or the shares of which are sold by the Trustee. The Trustee may vote such shares in its discretion.

Section 8. Express Powers of Trustee. Without in any way limiting the powers and discretions conferred upon the Trustee by the other provisions of this Agreement or by law, the Trustee is expressly authorized and empowered:

(a) To sell, exchange, convey, transfer, or otherwise dispose of any property held by it, by public or private sale. No person dealing with the Trustee shall be bound to see to the application of the purchase money or to inquire into the validity or expediency of any such sale or other disposition;

(b) To make, execute, acknowledge, and deliver any and all documents of transfer and conveyance and any and all other instruments that may be necessary or appropriate to carry out the powers herein granted;

(c) To register any securities held in the Fund in its own name or in the name of a nominee and to hold any security in bearer form or in book entry, or to combine certificates representing such securities with certificates of the same issue held by the Trustee in other fiduciary capacities, or to deposit or arrange for the deposit of any securities issued by the United States Government, or any agency or instrumentality thereof, with a Federal Reserve bank, but the books and records of the Trustee shall at all times show that all such securities are part of the Fund;

(d) To deposit any cash in the Fund in interest-bearing accounts maintained or savings certificates issued by the Trustee, in its separate corporate capacity, or in any other banking institution affiliated with the Trustee, to the extent insured by an agency of the Federal or State government; and

(e) To compromise or otherwise adjust all claims in favor of or against the Fund.

Section 9. Taxes and Expenses. All taxes of any kind that may be assessed or levied against or in respect of the Fund and all brokerage commissions incurred by the Fund shall be paid from the Fund. All other expenses incurred by the Trustee in connection with the administration of this Trust, including fees for legal services rendered to the Trustee, the compensation of the Trustee to the extent not paid directly by the Grantor, and all other proper charges and disbursements of the Trustee, shall be paid from the Fund.

Section 10. Annual Valuation. Commencing after initial funding of the trust, the Trustee shall annually, at least 30 days prior to the anniversary date of establishment of the Fund, furnish to the Grantor and to the

For Simple Use Only  
Comparable Wording Acceptable

appropriate EPA Regional Administrator a statement confirming the value of the Trust. Any securities in the Fund shall be valued at the market value as of no more than 60 days prior to the anniversary date of establishment of the Fund. The failure of the Grantor to object in writing to the Trustee within 90 days after the statement has been furnished to the Grantor and the EPA Regional Administrator shall constitute a conclusively binding assent by the Grantor, barring the Grantor from asserting any claim or liability against the Trustee with respect to matters disclosed in the statement.

Section 11. Advice of Counsel. The Trustee may from time to time consult with counsel, who may be counsel to the Grantor, with respect to any question arising as to the construction of this Agreement or any action to be taken hereunder. The Trustee shall be fully protected, to the extent permitted by law, in acting upon the advice of counsel.

Section 12. Trustee Compensation. The Trustee shall be entitled to reasonable compensation for its services as agreed upon in writing from time to time with the Grantor.

Section 13. Successor Trustee. The Trustee may resign or the Grantor may replace the Trustee, but such resignation or replacement shall not be effective until the Grantor has appointed a successor trustee and this successor accepts the appointment. The successor trustee shall have the same powers and duties as those conferred upon the Trustee hereunder. Upon the successor trustee's acceptance of the appointment, the Trustee shall assign, transfer, and pay over to the successor trustee the funds and properties then constituting the Fund. If for any reason the Grantor cannot or does not act in the event of the resignation of the Trustee, the Trustee may apply to a court of competent jurisdiction for the appointment of a successor trustee or for instructions. The successor trustee shall specify the date on which it assumes administration of the trust in a writing sent to the Grantor, the EPA Regional Administrator, and the present Trustee by certified mail 10 days before such change becomes effective. Any expenses incurred by the Trustee as a result of any of the acts contemplated by this Section shall be paid as provided in Section 9.

Section 14. Instructions to the Trustee. All orders, requests, and instruction by the Grantor to the Trustee shall be in writing, signed by such persons as are designated in the attached Exhibit A, or such other designees as the Grantor may designate by amendment to Exhibit A. The Trustee shall be fully protected in acting without inquiry in accordance with the Grantor's orders, requests, and instructions. All orders, requests, and instructions by the EPA Regional Administrator to the Trustee shall be in writing, signed by the EPA Regional Administrators of the Regions in which the facilities are located, or their designees, and the Trustee shall act and shall be fully protected in acting in accordance with such orders, requests, and

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instructions. The Trustee shall have the right to assume, in the absence of written notice to the contrary, that no event constituting a change or a termination of the authority of any person to act on behalf of the Grantor or EPA hereunder has occurred. The Trustee shall have no duty to act in the absence of such orders, requests, and instructions from the Grantor and/or EPA, except as provided for herein.

Section 15. Amendment of Agreement. This Agreement may be amended by an instrument in writing executed by the Grantor, the Trustee, and the appropriate EPA Regional Administrator, or by the Trustee and the appropriate EPA Regional Administrator if the Grantor ceases to exist.

Section 16. Irrevocability and Termination. Subject to the right of the parties to amend this Agreement as provided in Section 15, this Trust shall be irrevocable and shall continue until terminated at the written agreement of the Grantor, the Trustee, and the EPA Regional Administrator, or by the Trustee and the EPA Regional Administrator if the Grantor ceases to exist. Upon termination of the Trust, all remaining trust property, less final trust administration expenses, shall be delivered to the Grantor.

Section 17. Immunity and Indemnification. The Trustee shall not incur personal liability of any nature in connection with any act or omission, made in good faith, in the administration of this Trust, or in carrying out any directions by the Grantor or the EPA Regional Administrator issued in accordance with this Agreement. The Trustee shall be indemnified and saved harmless by the Grantor or by the Trust Fund, or both, from and against any personal liability to which the Trustee may be subjected by reason of any act or conduct in its official capacity, including all expenses reasonably incurred in its defense in the event the Grantor fails to provide such defense.

Section 18. Choice of Law. This Agreement shall be administered, construed, and enforced according to the laws of the State of Indiana.  
(name of state)

Section 19. Interpretation. As used in this Agreement, words in the singular include the plural and words in the plural include the singular. The descriptive headings for each Section of this Agreement shall not affect the interpretation or the legal efficacy of this Agreement.

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IN WITNESS WHEREOF, the parties below have caused this Agreement to be executed by their respective officers duly authorized and the corporate seals to be hereunto affixed and attested as of the date first above written.

By: A. E. Mollitt, Jr.  
(Signature of Grantor)

Vice President - Safety, Health  
and Environment  
(Title)

Attest:

R. J. Mosher  
Assistant Secretary  
(Title)

(SEAL)

Core States Bank, N.A.

By: Andrew C. Fisher  
(Signature of Trustee)

VICE PRESIDENT  
(Title)

Attest:

Anna G. Saxton  
Assistant Vice Pres.  
(Title)

(SEAL)

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Comparable Wording Acceptable

CERTIFICATE OF ACKNOWLEDGMENT  
FOR  
STANDBY TRUST FUND AGREEMENT

STATE OF Pennsylvania  
COUNTY OF Lehigh

On this 26<sup>th</sup> day of June, 1997, before me personally  
came A.E. Moffitt, Jr. to me known, who,  
(owner or operator)

being by me duly sworn, did depose and say that ~~she~~/he resides at  
Bethlehem Steel Corporation, Bethlehem, PA  
(address)

that ~~she~~/he is Vice President or Safety, Health  
(title)  
and Environment, Bethlehem Steel Corporation, the corporation  
(corporation)

described in and which executed the above instrument; that ~~she~~/he knows the  
seal of said corporation; that the seal affixed to such instrument is such  
corporate seal; that it was so affixed by order of the Board of Directors of  
said corporation, and that ~~she~~/he signed ~~her~~/his name thereto by like order.

Richard G. Masters  
(Notary Public)

NOTARIAL SEAL  
RICHARD G. MASTERS, Notary Public  
City of Bethlehem, Lehigh County, PA  
My Commission Expires Dec. 5, 2000  
Member, Pennsylvania Association of Notaries

(Seal)

SCHEDULE A

Identification of Facilities and Cost Estimates

Schedule A is referenced in the trust agreement dated June 26<sup>th</sup>, 1997

by and between Bethlehem Steel Corporation  
(name of owner or operator)

the "Grantor," and Core State Bank N.A.  
(name of trustee)

the "Trustee."

EPA identification numbers

Name of facility

Address of facility

Current plugging and  
abandonment cost estimate

Date of estimate

EPA identification number

Name of facility

Address of facility

Current plugging and  
abandonment cost estimate

Date of estimate

IN-127-1W-0001  
IN-127-1W-0003  
IN-127-1W-0004

Burns Harbor Division  
Bethlehem Steel Corporation  
U.S. Route 12

Westchester Township, IN 46304

\$421,700

June 12, 1997

## SCHEDULE B

**REFERENCE:** Trust Agreement, Section 3 - Establishment of Fund

**FACILITY:** Burns Harbor Division  
Bethlehem Steel Corporation  
U.S. Route 12  
Westchester Township, IN 46304

**EPA ID NO:** IN-127-1W-0001  
IN-127-1W-0003  
IN-127-1W-0004

DESCRIPTION OF PROPERTY		
<b>A. NEGOTIABLE SECURITIES</b>		
NAME OF ISSUE	FACE VALUE	MARKET VALUE
	\$	\$
SUBTOTAL		\$
<b>B. CASH</b>		
		\$
TOTAL AMOUNT OF COLLATERAL		

## **EXHIBIT A**

**REFERENCE:** Trust Agreement, Section 14 - Instructions to the Trustee

**FACILITY:** Burns Harbor Division  
Bethlehem Steel Corporation  
Burns Harbor, IN 46304

**EPA ID NO:** IND 003913423

All orders, requests, and instructions by the Grantor to the Trustee shall be in writing, signed by any person so designated in Section 5.03 of Article V of the By-laws of Bethlehem Steel Corporation, as amended October 1, 1988, to execute and deliver any contract or other instrument in the name of the Corporation and on its behalf. Section 5.03 of Article V appears as follows:

### **"ARTICLE V. CONTRACTS, CHECKS, DRAFTS, BANK ACCOUNTS, ETC."**

#### **SECTION 5.03. Execution of Other Contracts, etc.**

Except as otherwise required by law or by these By-laws, any contract or other instrument may be executed and delivered in the name of the Corporation and on its behalf by the Chairman, the President, a Vice Chairman, a Vice President, the Treasurer or the Commissioner; and the Board, by resolution; or the Chairman, the President, a Vice Chairman, a Vice President, the Treasurer or the Commissioner, by an instrument in writing filed with the Commissioner, may authorize any other officer or officers or agent or agents to execute and deliver any contract or other instrument in the name of the Corporation and on its behalf, and such authority may be general or confined to specific instances."



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
REGION 5  
77 WEST JACKSON BOULEVARD  
CHICAGO, IL 60604-3500

CC: HEM/WJK  
DET  
TWE✓

AUG 20 1997

CERTIFIED MAIL P-371-896-353  
RETURN RECEIPT REQUESTED

H. Roger Farber, Director  
Risk Management, Finance Department  
Bethlehem Steel Corporation  
1170 Eighth Avenue  
Bethlehem, Pennsylvania 18016-7699

ENVIRONMENTAL SERVICES	
SEP	7
WEI	WRE
OPE	DAM
KRB	TEL
RPH	PON
JNN	MLS
TLH	OLS
FILE	

REPLY TO THE ATTENTION OF:

WU-16J

Re: Finance Assurance Mechanism for the Underground Injection Control (UIC) Wells Located at the Burns Harbor Division, in Chesterton, Indiana

Dear Mr. Farber:

This letter is in response to your June 26, 1997, letter in which you asked to replace the current financial mechanism covering plugging and abandonment costs for the UIC wells at the Burns Harbor facility with a new financial mechanism. For the reasons specified below, the United States Environmental Protection Agency, Region 5, (USEPA) will not currently make this replacement. However, your request will be honored upon reissuance of UIC permits for these injection wells.

The UIC wells whose financial mechanism is now in question are the following:

Waste Pickle Liquor Well #1	Permit #IN-127-1W-0001
Waste Ammonia Liquor Well #1	Permit #IN-127-1W-0003
Waste Ammonia Liquor Well #2	Permit #IN-127-1W-0004
Galesville Sandstone Monitoring Well	No Permit

The permits for the first three of these wells reference the enforceable financial assurance mechanism at Parts I(H)(2) and III(D) in the permits. Part I(H)(2) specifies that the financial mechanism is provided in Attachment D of the permit, while Attachment D (Part III(D) of the permit) is a reproduction of a

funded Trust Agreement, whose funding began on July 31, 1986, and has been incrementally funded since.

In order to legally replace the financial mechanism currently in the permits (a funded Trust Agreement), Part III(D) in all the permits needs to be modified to replace the Trust Agreement with the new financial mechanism you provided in June 1997. As you may know, all three of the UIC permits have expired, but continue in force by virtue of Bethlehem Steel Corporation's timely submittal of permit renewal applications.

In a letter from Bethlehem Steel Corporation, signed by Superintendent John Sapia, and dated December 18, 1992, your corporation stated that it believed that the UIC permits expired and, as a result, cannot be legally modified. A copy of this letter is enclosed. Whether or not this legal interpretation is true, it certainly is at odds with the current need to modify these same expired permits.

Furthermore, the USEPA is currently actively engaged in reissuing all three of these permits. It is a waste of Agency resources to modify expired permits that are going to be imminently reissued. Upon reissuance of these permits, the USEPA will happily incorporate any adequate financial mechanism into the newly reissued permits. Hence, the current financial assurance mechanism can be released when the permits are reissued.

If you have any questions regarding this issue, please feel free to contact Nathan M. Wiser of my staff at (312) 353-9569.

Sincerely yours,



*for* Rebecca L. Harvey, Chief  
Underground Injection Control Branch

Enclosure

# Bethlehem Steel Corporation

BURNS HARBOR PLANT

BOX 248

CHESTERTON, IN 46304



December 18, 1992

Ref: LSW/DW/UIC

LSW0064

CERTIFIED MAIL  
RETURN RECEIPT REQUESTED

R. J. Zdanowicz, Chief  
Underground Injection Control (UIC) Section, WD-17J  
United States Environmental Protection Agency, Region V  
77 West Jackson Boulevard  
Chicago, Illinois 60604-3590

Subject: UIC Permit Nos. IN-127-1W-0003 and IN-127-1W-0004  
Bethlehem Steel Corporation, Burns Harbor Plant

Reference: Letter, R. J. Zdanowicz to J. P. Sapia, "Proposed Minor  
Modifications of Existing Underground Injection Control  
(UIC) Permits #IN-127-1W-0003 and #IN-127-1W-0004",  
dated November 17, 1992 (Received November 19, 1992)

Dear Mr. Zdanowicz:

This is to provide a timely response to the Reference letter.  
Bethlehem Steel cannot consent to the proposed modifications to the  
subject permits for the following reason.

Bethlehem believes that the subject permits have expired and,  
as a result, cannot be legally modified. As you know, the subject  
permits expired in September 1991 and currently remain enforce by  
virtue of Bethlehem's timely submittal of reapplications within 180  
days of the permit expiration date. Considering this, it is  
requested that draft renewal permits be issued that will  
incorporate the modifications noted in the Reference letter.

If there are any questions concerning this matter, please  
contact me or D. P. Bley at (219) 787-2712.

Very truly yours,

RECEIVED

DEC 22 1992

A handwritten signature in dark ink, appearing to read 'J. P. Sapia'.

J. P. Sapia, Superintendent  
Environmental Services Department

L. . .